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SEMI-ANNUAL REPORT

on

Essential Oils,
Synthetic Perfumes, &c.

Published by

SCHIMMEL & Co.

(Fritzsche Brothers)

MILTITZ

near Leipzig

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List of abbreviations.

d = specific gravity.
$d_{20°}$ = specific gravity at 20° (in references from our laboratories uniformly compared with water at 15°).
$d^{20°}_{4°}$ = specific gravity at 20°, compared with water at 4°.
$\alpha_{D20°}$ = optical rotation at 20°, in a 100 mm. tube.
$[\alpha]_D$ = specific rotation.
$n_{D20°}$ = index of refraction at 20°.
sol. p. = solidifying point.
m. p. = melting point.
b. p. = boiling point.
n = normal.
acet. = acetylation.
acid v. = acid value; ester v. = ester value; sap. v. = saponification value.
g. = gram; cc. = cubic centimeter; mm. = millimeter.

Temperatures are uniformly given in degrees Centigrade.
The strength of alcohol, if not otherwise indicated, is given in per cent. by volume.

Scientific and other Notes on Essential Oils.

Almond Oil, bitter. Not overmuch is yet known about the structure of amygdalin, the parent substance of volatile oil of bitter almonds. According to A. Bau's[1]) investigations, it does not contain any maltose, but a sugar $C_{12}H_{22}O_{11}$ which consists of 2 glucose components. He tested Saaz-yeast and *Saccharomyces Ludwigii*, Hansen on their behavior towards amygdalin. The fermenting experiments led to the result that amygdalase was present in the Saaz-yeast, but emulsin was totally absent. This may possibly explain why Saaz-yeast ferments amygdalin in a lesser degree than *Saccharomyces Ludwigii* which contains both amygdalase and emulsin. It does not contain any maltase, as it has no fermenting effect on maltose. As it, however, splits up amygdalin, one is driven to the conclusion that the sugar contained in amygdalin is not maltose, as mentioned above.

The fact that benzaldehydcyanohydrin belongs to the components of oil of bitter almonds which has not been deprived of its prussic acid induces us to draw the attention of the reader to a publication by K. Hess and K. Dorner[2]) on the alkyl ethers of this compound, of which the first part was issued some time ago.

In order to obtain the alkylated esters of benzaldehydcyanohydrin, the following means were adopted: —

1. Direct alkylation of the benzaldehydcyanohydrin.

2. In analogy with Favre's method for making chlormethylalkyl ethers which he specially worked out for formaldehyde: —

$$CH_2O + R\cdot OH + HCl = CH_2Cl(OR) + H_2O$$

the following process of formation from benzaldehyde, hydrochloric acid and the corresponding alcohol would seem to be possible: —

$$C_6H_5\cdot CHO + HCl + R\cdot OH \longrightarrow C_6H_5\cdot CH(Cl)(OR) \xrightarrow{KCN} C_6H_5\cdot CH(CN)(OR).$$

3. Starting from phenylchloracetic acid this might be transformed by means of sodium alcoholates into alkylated mandelic acids, the carboxyl group substituted by the carbamide group and finally the following type obtained by dehydration: —

$$C_6H_5\cdot CH(Cl)COOH \longrightarrow C_6H_5\cdot CH(OR)\cdot COOH$$
$$\longrightarrow C_6H_5\cdot CH(OR)\cdot CO\cdot NH_2 \longrightarrow C_6H_5\cdot CH(OR)(CN).$$

Of these 3 methods the authors found the last to be the most suitable and according to this process they prepared the methyl and ethyl derivatives of benzaldehydecyanohydrine.

The *O*-methylmandelic nitrile (α-methoxyphenylacetonitrile) is a mobile, refractive and very stable oil with a rather pleasant smell, reminding of benzaldehyde, which, however, becomes rather pungent when its vapour is concentrated. Boiling-point 116 to 118° (14 mm.). When heated with dilute sulphuric acid it is saponified into the methoxy acid.

The *O*-ethylmandelic nitrile (α-ethoxyphenylacetonitrile) boils between 122 and 124° (16 mm.) and is a viscid, transparent, stable oil with rather an aromatic odour.

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[1]) *Perfum. Record* 7 (1916), 303. — [2]) *Berl. Berichte* 50 (1917), 390.

Oil of an Andropogon Species. The leaves and roots of an andropogon grass, found in the Serbian district of Semendria, which apparently seems to be either *Andropogon Gryllus,* L. or *A. Ischœmum,* Thunb., produced in a trial distillation we attempted 0.3 per cent. of a light yellow oil, having a curious rough, not very pleasant odour. The constants observed were; $d_{15°}$ 0.9680; α_D — 10° 52′; $n_{D20°}$ 1.49951; acid v. 1.9; ester v. 8.4; soluble in about 2.5 vol. of an 80 per cent. alcohol. It does not resemble any of the known andropogon oils and is not suitable for perfumery purposes. Camphor, as indicated by the smell of the grass, was not present in the oil.

The roots were forwarded to us from the Agricultural Botanical Institute at Munich and are used at Semendria for the manufacture of rootbrushes.

Angelica Oil. On the cultivation of angelica, comp. p. 110.

Aniseed Oil. A so-called *Oleum anisi vulgaris,* offered us for purchase, proved on investigation to be a light fennel oil, such as is obtained as a by-product in the manufacture of anethol. Its optical rotation was + 33° and even in a refrigerating mixture it did not deposit any anethol on addition of an anethol crystal, so that this body can only have been present in quite minimal quantities, whereas the principal constituent was fenchone.

We mention the matter in this place in order to caution would-be buyers of aniseed oil.

Oil of Artemisia annua. The oil of the composita *Artemisia annua,* L. is still little known[1]). Yoshikazu Imada[2]) now throws some light on its composition. Phellandrene, pinene, limonene, and dipentene were not contained in the oil, on the contrary however cineole and a compound $C_{10}H_{16}O$, which formed a semicarbazone melting between 95 and 96°. The oil examined by Imada showed the constants: $d_{15°}$ 0.8984; α_D — 16.71°. The oil we examined and distilled ourselves gave at the time the following results: $d_{15°}$ 0.8912; α_D — 1° 18′.

Oil of Artemisia tridentata. As we have already remarked, eucalyptus oil is used in Australia in enormous quantities in foundries for flotation purposes[3]). The oil of *Artemisia tridentata,* Nutt., N. O. *Compositæ*[4]), is also remarkably suitable for this purpose, as M. Adams[5]) found on the occasion of an examination of various volatile oils executed in the laboratory of the University of Nevada. The plant is widely distributed in the West, especially in western Missouri. The oil is produced by means of distillation with steam under high pressure; the yield varies according to the time of year from 0.4 to 1.0 per cent. The crude oil is dark in colour; when again distilled with steam, as before, it is at first quite colorless, but becomes straw-coloured on standing. Statements are further made concerning other physical properties, but unfortunately there are no particulars thereon in the abstract. The oil contained a small quantity of α-pinene and β-pinene, but the bulk smelled distinctly of camphor.

G. H. Clevenger[6]) gives particulars on the experiments made by various authors tor the object of producing the oil of various species of sage *(Artemisia?)* and for

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[1]) Comp. Gildemeister und Hoffmann, *Die ätherischen Öle,* 2nd ed., vol. III, p. 696. — [2]) Yakugakuzasshi; *Chemist and Druggist* **89** (1917), 376. — [3]) *Report* October **1911**, 49. — [4]) The plant is referred to in the abstract as sage or black sage. In America, the Labiata *Ramona Stachyoides* is also called "Black sage". — [5]) *Bll. Am. Min. Eng.* **1916**, 1685; *Zeitschr. f. angew. Chem.* **30** (1917), II. 129. — [6]) *Bll. Am. Min. Eng.* **1916**, 1685; *Zeitschr. f. angew. Chem.* **30** (1917), II. 129.

festing the oil with the object of examining its applicability for flotation purposes. The oil produced from "black sage" has a certain resemblance to the eucalyptus oil found in the Australian *Eucalyptus amygdalina*. A considerable quantity of oil from *Artemisia tridentata* was divided into fractions which were tested for their fitness for the flotation of various minerals, such as galena, pyrites &c. A tar oil fraction appeared to be specially suitable for this purpose, with which quantitative experiments were made on zinc, quicksilver, lead and gold-silver ores; the abstract, however, gives no particulars as to the results obtained.

According to G. D. van Arsdale[1]), the various oils do not act in the same sense when applied for flotation purposes. One group of them is suitable for forming a thin coating over the particles of the sulphides; these are called "oilers", the others "foamers". The latter produce the froth and thereby reduce the surface tension of the water. The froth only acts by increasing the surface and is, in consequence, dispensable. When forming an emulsion, an oil becomes ineffective. The use of sulphuric acid leads to the disappearance of any emulsion which may have been formed. The favourable effect of the addition of acids must be attributed to this quality. Instead of mixing the oils at the outset, it is much more advantageous first to add the "oilers" and then to run the "foamers" in, when the former have enveloped the particles of the sulphides.

According to H. J. Stander[2]), the intermediate surface tension between water and particles of sulphide is increased by the addition of oil and its bearing power is enhanced. Whereas pine tar drives the colloids into the froth, coal tar does not have this effect. Probably the particles of sulphide are charged with positive electricity in the flotation process, those of quartz with negative electricity.

Bay Oil. Besides the genuine bay tree a variety, *Pimenta acris* var. *citrifolia* is to be found in the West Indian Islands, which, owing to the lemon-like smell of its leaves, is termed "Lemoncilla", "Bois d'Inde citron" or "False bay" and whose leaves are also called "Citronella leaves". The oil derived from them contains large quantities of citral and cannot really be termed bay oil[3]).

It goes without saying that in starting a plantation of bay trees attention must be paid to the fact that this inferior variety is not included amongst the other plants. This error, however, was committed[4]) in the trial plantation on the Isle of Tortola; in fact it was proved at a recent examination that 35 per cent. of the seedlings there present belonged to the lemon-like-smelling variety. It appears that the mother plant was a hybrid. The leaves looked quite normal and nothing but their odour betrayed the fact that they belonged to the inferior species.

Betel Oil. On page 32 of our April *Report* **1914** we published an investigation by Mann, Sahasrabudha and Patwardhan on the physiology and chemistry of betel leaves which are in general use in India for chewing purposes. In a recent publication, H. H. Mann and V. G. Patwardhan[5]) referred again to this subject. The betel leaves suitable for chewing are generally mature or over-mature and not yet brittle. In leaves which are not yet mature and are still growing, the percentage of starch, non-reducing

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[1]) *Eng. Min. Journ.* **101** (1916), 851; *Zeitschr. f. angew. Chem.* **30** (1917), II. 129. — [2]) *Eng. Min. Journ.* **101** (1916), 756; *Zeitschr. f. angew. Chem.* **30** (1917), II. 129. — [3]) Comp. Gildemeister und Hoffmann, *Die ätherischen Öle*, 2nd ed., vol. III, p. 204. — [4]) *Perfum. Record* 7 (1916), 303. — [5]) *Mem. Dep. Agric. India* 4 (1916), 281; *Journ. Soc. chem. Industry* **35** (1916), 1033.

sugars, and nitrates increases during day-time, whereas that of reducing sugars whose quantity is two-thirds of the total sugars, besides that of tannin and oil, remains fairly constant throughout the day. Over-mature leaves contain much less reducing sugars, whereas the proportion of non-reducing sugars, which are decidedly predominant, shows little change. Leaves suitable for chewing must contain little starch and reducing sugars, but large quantities of sucrose. The most important factor in forming an estimate of the value of the leaves is the amount and nature of the volatile oil present. The more oil the leaves contain, the better they are for chewing; over-mature leaves contain less oil. The volatile oil of the betel leaves (*Pan* oil) is generally known to consist of phenol and terpene compounds in the main, the relative proportions of which vary in the different species of leaves; those leaves containing most phenol are the best. The proportion of phenols varies in the non-bleached leaves from 42 (*Poona* green leaves) to 70 per cent. (*Ramtek kapuri* leaves), and in the dark-green leaves from 39 to 45 per cent. Bleaching increases the percentage of oil and phenol. The bleaching process itself is best done in the dark; the surrounding atmosphere must be damp to prevent the leaves from getting too dry, but the moisture must not be excessive, otherwise the leaves tend to rot. The temperature of the bleaching room must be between 29 and 33°, temperatures over 35° are harmful, under 24° useless. If the bleaching is continued beyond producing a whitish-yellow appearance of the leaves, the quality of the leaves and their oil content suffers.

Calamus Oil. We occasionally received small lots of fresh calamus roots from the low-lying districts near the Elster and Saale rivers, so that we were enabled to fill up our stocks a little. Unfortunately, it was impossible to buy large quantities of the roots, owing to the scarcity of men capable of gathering them.

Amongst the calamus oil samples tested we again found several which were considerably adulterated. An oil with the constants, $d_{15°}$ 0.9715; $\alpha_D + 11° 6'$; acid v. 0.9; ester v. 157.7 had suffered, *inter alia,* an addition of glycerol acetate. Accordingly, it was extracted from the oil by repeated agitation with water and proved by its high ester value of 700 to be triacetin.

In another case the adulterant was terpineol. This was betrayed by the too high acetylation value and the strikingly good solubility, whereas for the rest the degree of rotation was comparatively low, *viz.,* $d_{15°}$ 0.9617; $\alpha_D + 7°$; acid v. 1.8; ester v. after acet. 87.7; soluble in 1 vol. and more of 80 per cent. alcohol. By way of explanation we may add that normal calamus oils do not dissolve clearly even in 10 vol. of 80 per cent. alcohol and that their acetylation value varies between 30 and 50. On fractionating the adulterated product a fraction was obtained which smelled strongly of terpineol, which then was identified by its nitrosochloride.

In another case the adulteration was not perceptible from the constants ($d_{15°}$ 0.9702; $\alpha_D + 10°$; acid v. 1.8; ester v. 5.6; soluble in any vol. of 90 per cent. alcohol); the oil was principally remarkable owing to its weak and at the same time striking smell, and the suspicion that the cause was an adulteration was proved to be right by further investigation. In fact these methods succeeded in isolating about 10 per cent. safrole from the oil which was easily recognized by its characteristic smell and by the fact that, when cooled down and inoculated with a little solid safrole, it immediately became solid. As safrole by no means belongs to the components of calamus oil, its detection makes the adulteration self-evident, although apparently not pure safrole was employed, but a camphor oil fraction containing safrole.

Oil of Calycanthus occidentalis. *Calycanthus (Butneria) occidentalis,* Hook. et Arn. (N. O. *Calycanthaceæ*) is a shrub found in northern California and south Oregon which is called "Spice Bush". The composition of the oil resembles that of *Calycanthus floridus*, which Miller investigated some time ago[1]). C. C. Scalione[2]) obtained from the leaves 0.15 per cent. and from the twigs 0.37 per cent. of a greenish-yellow volatile oil having a camphor-like smell and bitter taste. Constants: $d_{20°}$ 0.9295; $\alpha_D + 7° 28'$; $n_{D20°}$ 1.4713; acid v. 0.05; sap. v. 54.3; ester v. after acet. 33.5. The composition was about the following: 50 per cent. cineole, 8 per cent. *d*- and *l*-pinene, 9 per cent. borneol, 19 per cent. linalyl acetate, besides small quantities of camphor, methyl salicylate, and sesquiterpene alcohols. How the presence of these compounds was proved does not appear from the abstract. The oil dissolves in all proportions in 90 per cent. alcohol and in 15 to 16 vol. of 70 per cent. alcohol.

Camphor Oil. As the American Consul-General in Yokohama reports[3]), an extraordinary rise is noticeable in the Japanese production of camphor, as the demand increases, on the home market as well as on foreign ones, with the continuation of the war. In Formosa, great efforts were made in order to foster the cultivation of camphor trees. In 1916, 528000 lbs. of camphor on an average were shipped monthly to the U. S. A., and owing to a new agreement between the Japanese Government and American dealers shipments have risen to more than 1 million lbs. after April 1st. According to the calculations of the *Japan Gazette*, the output of camphor in Formosa, in 1916, amounted to 11.6 million lbs. to the value of 3.2 million dollars. Camphor prices show a continuous upward tendency, so that the Government counts upon still higher revenues.

According to W. A. Dyes[4]), the export of camphor oil from Japan has, it is true, increased after the establishment of refineries in Formosa, but in contradiction to the statement given before the export of camphor has decreased.

In 1913, the value of the exports of camphor from Japan was 2235784 yen; in 1914, 2780001 yen. The exports went to the following countries[5]): —

Camphor	1913	1914
	in 1000 yen	
British India	830	824
Great Britain	137	332
France	67	224
Germany	426	260
U. S. A.	434	696
Australia	102	104
other countries	237	336
total:	2235	2780.

Germany imported from Japan about 4900 piculs in 1912 and 1913 and, in 1912, 16700 piculs from Formosa to the value of 1573000 yen, whereas the quantity fell to 7925 piculs to the value of 696000 yen in 1913. As per above statement, a large part went to British India, where camphor is strown upon Buddhist holocausts for purifying purposes. As for this purpose the cheap refuse camphor and camphor

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[1]) *Journ. Americ. chem. Soc.* **36** (1914), 2182; *Report* April **1915**, 9. — [2]) *Journ. ind. eng. Chemistry* 8 (1916), 729; *Journ. Soc. chem. Industry* **35** (1916), 978. — [3]) *Zeitschr. f. angew. Chem.* **30** (1917), III. 341. — [4]) *Chem. Ztg.* **41** (1917), 518. — [5]) Comp. *Report* October **1916**, 9.

powder are quite good enough, these qualities are imported into India in increasing quantities, thus supplanting the refined product.

The production in Formosa amounted to: —

3.116 million kilos in 1910, 2.7 million kilos in 1911, 2.4 million kilos in 1912.

The output of refined camphor in Japan itself was: —

2319 million kins to the value of 2029 million yen in 1913/14,
3114 „ „ „ „ „ „ 2867 „ „ „ 1914/15.

Formosa exported for: —

5.529 million yen in 1910, 4.856 million yen in 1911, 5.814 million yen in 1912.

The production and sale of camphor oil in Formosa showed the following figures, respectively: —

in 1910: 3.31 million kilos, in 1911: 3.18 million kilos, in 1912: 3.71 million kilos,
„ 1910: 3.96 „ „ „ 1911: 1.91 „ „ „ 1912: 1.21 „ „ .

The chief quantities of camphor and camphor oil in Formosa are manufactured in private refineries (?) which belong to 6 Japanese and 9 natives, respectively, and they in turn control 5753 small producers. About 9000 workers are employed in collecting the raw material and in extracting the camphor. The crude camphor is taken over and refined by the Government. The export for the Government is effected by the Mitsui Bussan.

When the monopoly was introduced in Formosa, in 1899, it was thought that it practically meant the control of the world's market; but as owing to the monopoly the prices went up, the production in Japan and South China was fostered and encouraged. The output in the private forests of Japan rose very much, owing to destructive exploitation regardless of the future, the tree-stumps and roots being dug up and extracted. At present there are scarcely any camphor trees left in private hands in Japan. The number of trees existing there is estimated at 10 millions, the yield of which is considered to come up to about 2.4 million cubic feet (?). In Formosa, the camphor trees not under 100 years old are supposed to represent 27 million (?) cubic feet which ought to yield 19.2 million (?) kilos of camphor and 14 million kilos of camphor oil. As the trees must be more than 60 years old before they yield reasonable quantities of camphor, it is possible that, unless due care be taken, within a few decennaries no mature trees will be left.

American commercial quarters consider the fact that the shipments of crude camphor to the U. S. have been stopped as a proof of the Japanese Government's intention to use the monopoly in order to foster the Japanese refineries at the cost of those in other countries. In 1916, the North American refineries held very small stocks of crude camphor, so that they had to buy the refined product in order to fill their contracts. No official explanation was given by Japan as to why the supplies of crude camphor to the U. S. were suspended. Needless to say, it is possible that Japan means to extend its monopoly also to the refined product, so as to obtain a complete control of this branch of commerce. Taking into consideration Japanese methods of fostering the home industries, it is probable that this way was chosen in order to cause an extension of the refineries.

The production of camphor in Japan in the year closing on March 31st, 1917 is estimated at 1627422 kins: an increase of 26607 kins, as compared with the production of the year 1915/16. The estimation regarding Formosa amounts to 5014743 kins, representing an increase of 394561 kins above the quantity of the preceding year.

The production of camphor oil in Japan is estimated at 3210494 kins for 1916/17, *i. e.* 209073 kins more than the actual quantity of the year 1915/16; in Formosa, it might come to 7827560 kins, or 946328 kins more than in the preceding year[1]).

In other quarters, the production of camphor in Japan in 1916/17[2]) is estimated at 1.92 million kilos, or 120000 kilos more than in 1915/16, and, in Formosa, at 4.68 million kilos, or about 570000 kilos more than in 1915/16. In 1916, the U. S. imported from Kobe still more camphor than in 1915. The figures were: —

2214547 lbs. to the value of $ 1100694 in 1916,
2172972 „ „ „ „ „ „ 454016 „ 1915.

As, according to another newspaper report, synthetic camphor may not be exported, the export of Formosa camphor, chiefly to England, the U. S. and British India, has risen considerably. During the first 9 months of 1916, the value of the camphor exports from Formosa came up to 3964000 yen against 2508000 yen for the same period in the preceding year[3]).

We have, of late, been asked from different quarters to examine samples of so-called synthetic camphor, which all proved to be borneol. This could be deduced already from the smell of the product and the shape of the crystals and could be proved by determining the m. p., which was between 203 and 204°, whereas camphor melts between 175 and 179°. The optical rotation, determined in one case, was $[\alpha]_D + 30° 23'$ (in a 32 per cent. alcoholic solution), which shows that this borneol chiefly consisted of the dextrorotatory modification. On acetylation of the same product, it was shown that it did not contain any other components in addition to borneol. A 21.2 per cent. solution in xylene showed, after acetylation, an ester v. of 74.7, corresponding to a content of 21.8 per cent., a figure which tallies very well with the concentration of the solution and characterizes the product as pure borneol.

Even nowadays, a rather easy conscience is necessary to sell borneol as synthetic camphor. Although there is a certain resemblance in the medical action of the two preparations, it is not great enough to permit of substituting the one for the other; on the contrary, especially if internal treatment is concerned, such a proceeding might have rather disagreeable consequences.

Perhaps all the products submitted to us come from the same source. In one case, we were able to find out that the seller was a pharmaceutical chemist. On being asked for an explanation, he declared that he bought the substance as synthetic camphor and considered it as such, it might perhaps not give all the reactions required by the German Pharmacopœia, but this was due to present conditions. This is, indeed, a short answer, but it throws a peculiar light on the scientific proficiency of the pharmaceutical chemist in question, for a simple determination of the m. p. ought to have convinced him of the fact that what he sold was by no means identical with synthetic camphor. We can only hope that this lack of judgment with regard to a pharmaceutical product will not have unpleasant censequences for him.

That borneol has been offered and sold as camphor, has been also stated in other quarters[4]).

The fact that, owing to the war, the demand for camphor is very great and that its synthetic preparation has come to the front again, as the natural product is no

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[1]) *Board of Trade Journal; Nachr. f. Hand., Ind. u. Landw.* **1917**, N° 46, p. 4. — [2]) W. A. Dyes, *Chem. Ztg.* **41** (1917), 519. — [3]) *Tropenpflanzer* **20** (1917), 247. — [4]) *Pharm. Ztg.* **62** (1917), 475.

longer accessible for us, induced P. Bohrisch[1]) to publish an article on natural and synthetic camphors. Although the subject is of great interest just now, we may limit ourselves just to mentioning the article, as it does not contain anything new of importance.

Doubtless camphor oil belongs to the most carefully examined volatile oils and numbers of its components have been verified. That, however, this oil still often contains many components which hitherto have escaped observation, is an obvious supposition. Hence it is not surprising that recently K. Kafuku[2]) should have proved the existence of laurinic acid in a sample of oil, distilled by the Camphor Monopoly Office at Formosa. This oil showed the following constants: $d_{15°}$ 0.9805; $n_{D17.5°}$ 1.5035; acid v, 3.14; ester v. 0.66; ester v. after acet. 126. Besides this the oil contained a compound $C_{14}H_{26}O_2$, being probably the lactone of an aliphatic hydroxyacid, as well as a tricyclic sesquiterpene alcohol. In the highest-boiling fractions piperonylic acid was present which perhaps was formed during distillation by the oxidation of safrole. The abstract, however, gives no particulars as to the characteristics and properties of these compounds.

Cardamom Oil. In the annual report of the Matale Planters' Association of Ceylon for 1916[3]), the weather is called unfavourable for the development of the cardamoms, being too seldom damp and misty, as the plants require. The crop is somewhat smaller than that of 1915, as new fresh plantations have been started, whereas many old fields show signs of exhaustion. In Colombo, the demand relaxed at times, but became a little more vivid towards the close of the year. It was likewise difficult in London to sell important lots, although the prices were rather satisfactory during the first half of the year. In the second half of the year, the demand rose from the U. S., but the prices went down somewhat. There are 1664 acres under cultivation, 1440 of which are productive. A crop of about 680 000 lbs. is expected for 1917.

In the report of the Ceylon Planters' Association, the exports in 1916 are estimated at 427 843 lbs., against 519 039 lbs. in 1915. No doubt, considerable quantities are being held back, owing to the low prices and in the expectation of better ones later on. The prices for green-dried as well as for fully cured cardamoms are so low that it hardly pays to grow them. The shipments in 1917 will not exceed 500 000 lbs.

Cedar Oil, Himalayan. Under this heading we recently[4]) described an oil which the Forest Research Institute and College at Dehra Dun had sent us for examination and which had been distilled from the wood of *Cedrus Deodara,* Loud. *(C. Libani,* Barr. var. *Deodara).* This wood is used in India for building purposes, incidentally also for railway sleepers. It has been proposed to distil the refuse in order to utilize it; in the same manner cedar wood chips are treated in the manufacture of pencils. O. D. Roberts[5]), who investigated the oil closely, draws attention to the fact that the botanical status of the Himalayan cedar ("Deodar Tree") is still uncertain. Some authors regard it as a definite species (*Cedrus Deodara,* Loud.), whereas others take it for a variety of *C. Libani.*

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[1]) *Pharm.Zentralh.* **57** (1916), 683, 699. — [2]) *J. chem. Ind., Tokyo* **19** (1916), 815; *Journ. Soc. chem. Industry* **35** (1916), 1178. — [3]) *Chemist and Druggist* **89** (1917), 382. — [4]) *Report* April **1915**, 52. — [5]) *Journ. chem. Soc.* **109** (1916), 791.

Roberts received both the oils he examined from the Forest Economist to the Government of India. The constants of the products examined by him and by us were the following: —

	Himalayan Cedar Oil tested by		
	Roberts		Schimmel & Co.
	I (received 1914)	II (received 1915)	
$d_{15°}$	0.9549	0.9756	0.9530
α_D	+ 52° 16′	+ 34° 6′	+ 53° 8′
$n_{D21°}$	1.5195	1.5225	$n_{D20°}$ 1.51565
acid v.	5.6	4.5	2.9
ester v.	19.3	4.9	14.9
ester v. after acet.	30.8	34.4	39.2
	not soluble in 90 per cent. alcohol.	soluble in all proportions in 90 per cent. alcohol.	not completely soluble in 90 per cent. alcohol.

With the help of the semicarbazone (melting point 163 to 164°) Roberts succeeded in obtaining a ketone $C_9H_{14}O$ from the lower-boiling fractions which gave no reaction with alkali sulphites. The oxime remained liquid, but produced with bromine a dibromoxime melting at 130°. The smell of the ketone recalls that of amylacetate; it is probably identical with p-methyl-Δ^3-tetrahydroacetophenone which Stephan and Helle[1]) obtained in our laboratory by oxidizing β-terpineol. On splitting the semicarbazone with dilute sulphuric acid the original ketone was not regenerated, but the main product that appeared was a ketone boiling between 222 and 224° ($n_{D20°}$ 1.4965) whose semicarbazone melted between 204 and 205° and which was identical with *p*-tolyl-methylketone, as on oxidation with alkaline bromine solution it produced bromoform and *p*-toluic acid. Probably the ketone $C_9H_{14}O$, found by Grimal[2]) in Atlas cedar oil, is also identical with *p*-methyl-Δ^3-tetrahydroacetophenone. The Himalayan cedar oil contained besides small quantities (about 0.1 per cent.) of a phenol which was identified by its benzoyl compound, melting at 70°. Roberts found in the saponification liquors of the oil a mixture of caproic, œnanthic (analysis of the silver salts) and stearic acids (m. p. 71°; analysis of the silver salt). The chief constituents of the oil were sesquiterpene compounds, amongst which Roberts isolated a sesquiterpene boiling between 262 and 265° (151 to 153° at 19 mm. pressure): $d_{15°}$ 0.9276; $\alpha_{D20°}$ + 14°36′; $n_{D21°}$ 1.5170. It produced in a solution of glacial acetic acid with concentrated sulphuric acid the same coloration as cadinene. No solid derivatives were obtained from the sesquiterpene.

We[3]) had also found a ketone in the Himalayan cedar oil which we considered as possibly identical with the ketone originating from Atlas cedar oil, as well as a sesquiterpene not identical with cadinene.

Cherry Kernel Oil. A few years ago already we described an oil we had obtained by distilling cherry kernels[4]). An oil showing quite different qualities has been described by F. Rabak[5]) in a paper on the utilization of cherry by-products. In the cherry preserving industry of North America about 1400 tons of cherry stones and 112000 gallons of juice are obtained as by-products; hitherto these products have not been utilized, although this is quite possible. By treating the crushed stones with a solvent, 8.3 per cent.

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[1]) *Berl. Berichte* **35** (1902), 2151. — [2]) *Compt. rend.* **135** (1902), 582, 1057; *Report* April **1903**, 18. — [3]) *Report* April **1915**, 52. — [4]) *Report* April **1913**, 111. — [5]) *U. S. Dept. Agric. Bull.* N° 350, 1916. — *Bull. Agr. Intell.* **7** (1916), 1355; *Journ. Soc. chem. Industry* **36** (1917), 159.

of a fatty oil can be obtained; if the kernels are pressed without the shells, even 30 per cent. are obtainable; the press-cake yields about 1 per cent. of a volatile oil.

The qualities of the fatty oil are the following: —

	$d_{25°}$	$n_{D25°}$	solidification point.	sapon. v.	iodine v.
Oil produced from whole stones . .	0.9137	1.4641	+ 12°	179.7	93.7
Oil from kernels freed from shells .	0.9092	1.4635	+ 13°	180.8	92.8

The volatile oil gave the following results: $d_{4°}$ 1.012; it contained 7.94 per cent. prussic acid and 67.95 per cent. benzoic acid. The product we distilled showed the following characteristics: $d_{15°}$ 1.0532, and contained 0.27 per cent. of prussic acid. Accordingly the oil described by Rabak showed very different qualities to that we produced; we are inclined to suspect, however, that the statement, according to which Rabak's oil contained 67.95 per cent. of benzoic acid, must be attributed to a misprint, or possibly it was a case of a very strongly oxidized oil which can not have been liquid at ordinary temperature.

Cherry Laurel Oil. According to A. Saint-Sernin[1]), it is useless to reduce the leaves of cherry laurel into small pieces in order to prepare cherry laurel water. From 60 kg. of leaves which had not been cut up beforehand and were macerated for 16 hours, he obtained 60 litres of cherry laurel water with a prussic acid content of 0.144 per cent.; another trial without maceration produced 0.123 per cent. of prussic acid.

Navarre[2]) obtained from 1314 kg. of macerated leaves 1314 litres of cherry laurel water with 0.1288 per cent. prussic acid. This obliges one to conclude that maceration is in point of fact useless; as, however, M. Bridel and N. Delabrière[3]) remark, this question can only be solved by comparative tests with leaves from the same tree and gathered at the same time. With this object in view they collected on Jan. 13th, 1915, 20 kg. of leaves from the same tree and divided them into portions of 2 kg. each. Of these various parcels of leaves,

the first parcel was thrown into a distilling apparatus without being cut up at all;
the second was also not cut up, but macerated for 16 hours and then distilled;
the third parcel was cut up with a pair of scissors into narrow strips and immediately distilled;
the fourth parcel was disintegrated in a machine[4]) and then immediately distilled.

These 4 tests were made on the same day the leaves were gathered. On the following day 4 more parcels were treated in the same manner and finally on the third day the remainder was collected, disintegrated, and immediately distilled.

The authors used for each 2 kg. of leaves 8 kg. of distilled water.

The results of the trials were as follows: —

Cherry laurel water produced from:	Prussic acid content: on the 1st day	on the 2nd day	3rd day.
Whole leaves without maceration	0.0918 per cent.	0.0817 per cent.	—
„ „ after „	0.0931 „ „	0.0999 „ „	—
Leaves cut up by scissors . . .	0.1201 „ „	0.1188 „ „	—
„ „ „ in a machine . .	0.1701 „ „	0.1552 „ „	0.1644 per cent.

Disintegration of the leaves produces evidently a rise in the prussic acid content.

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[1]) *Bull. des trav. de la Soc. de Pharm. de Bordeaux* **52** (1912), 431; *Journ. de Pharm. et Chim.* **6** (1912), 511. — [2]) *Ibidem* VII. **3** (1911), 15. — [3]) *Ibidem* VII. **11** (1915), 110. — [4]) As to the construction of this machine, comp. M. Bridel, *ibidem* VI. **26** (1907), 21.

The duration of the distilling process has also some effect on the production of prussic acid; leaves which were thrown into boiling water and then immediately distilled gave a cherry laurel water only containing 0.0175 per cent. of prussic acid.

In connection with these facts we report on some directions by O. Rössler[1]) on the production of cherry laurel water which according to this author might quite well be employed instead of bitter almond water. North Africa and the South of France are our principal purveyors for bitter almonds. As, however, we are prevented under present circumstances from obtaining them from these countries, we are compelled to use substitutes in the shape of our indigenous plants, unless we follow the example of the English and Americans by using diluted prussic acid instead of bitter almond water.

The cherry laurel, *Prunus Laurocerasus,* L. (N. O. *Rosaceæ*) is widely disseminated in the South and South-west of Germany as a tree and bush and could easily, according to Rössler, supply all our demands for a substitute for bitter almond water. Rössler gathered from the literature he had at hand that cherry laurel water had always played a part in the medical works of Southern Germany and further that as a concession towards South-German ideas it had been accepted by the first German Pharmacopœia, a position it had, however, subsequently to relinquish in favour of bitter almond water.

As a result of his investigations Rössler is inclined to recommend the following method for producing cherry laurel water: —

One part of cherry laurel leaves from the oldest trees available is gathered in July or August, cut up, macerated, and, after mixing with 3 parts of water, is left standing in a retort for 24 hours, whereupon $^3/_4$ of the mixture is distilled into a receiver, containing 0.2 parts of alcohol, air being excluded during the process. The residue of the leaves is distilled again after a period of 3 weeks, yielding one-half part of a second running for standardizing the first runnings. In case the percentage of prussic acid contained in cherry laurel leaves of German origin were less than that usually found in the foreign product, one might consider the question of standardizing the palpably very aromatic distillate by the addition of benzaldehyde-cyanohydrine. In any case, a really aromatic smelling and tasting cherry laurel water, prepared in this manner, would be infinitely preferable to the artificial products usually put on the market.

Cinnamon Oil. A critical discussion of the testing methods adopted by the German Pharmacopœia, 5th ed., is to be found on page 83.

Oil of Cinnamomum Oliveri. *Cinnamomum Oliveri,* Bail. (N. O. *Lauraceæ*) ("Brisbane Sassafras") is a handsome tree with grey, rough bark which is to be found in the "Scrubs" of Queensland, also of New South Wales. The bark oil has already been described a number of years ago by H. G. Smith, an account to which R. T. Baker[2]) referred at the time. Smith published the following list of components: cineole, eugenol (?), cinnamaldehyde (?) and a stearoptene which separated on cooling to —12°. G. W. Hargreaves[3]) arrived at quite different results, when he recently investigated the oil thoroughly. He could find no trace either of cineole, cinnamaldehyde, nor of eugenol. Nor could he verify Smith's statement, according to which the oil contained no low-boiling terpenes. One is inclined to believe that the original plant of both oils differed, one must,

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[1]) *Arch. der Pharm.* **255** (1917), 151. — [2]) Comp. Gildemeister and Hoffmann, *The Volatile Oils,* 2nd ed., vol. II, p. 440. — [3]) *Journ. chem. Soc.* **109** (1916), 751.

however bear in mind, as Hargreaves remarks, that Smith only identified the components by colour reactions or by their smell and that he neither made analyses nor produced derivatives.

Hargreaves obtained from the bark 2.47 per cent. of an oil with the following constants: $d_{23°}$ 1.030; $n_{D23°}$ 1.5165, which he separated by repeated fractionation (30 mm. pressure) into components boiling up to 90°, from 90 to 120°, from 120 to 135°, and from 135 to 155°. In the first fractions he proved the presence of α-pinene by the nitrosochloride, melting at 108°. The second fraction contained *d*-camphor (boiling point 204°; oxime, melting at 119°). In the third fraction safrole was present (melting point 8°; constants: $d_{18°}$ 1.09; $n_{D20°}$ 1.535) which was characterized by the preparation of its α-nitrosite melting between 129 and 130°. The highest-boiling fraction contained methyl eugenol which boiled between 146 and 147° at 30 mm. pressure, and at 244° at ordinary pressure, forming veratric acid (melting point 179°), when oxidized with potassium permanganate, and formed a tribromo derivative, melting between 77 and 78°. The fractions boiling above 150° (30 mm.) appeared to consist in the main of methyl eugenol, as on being oxidized with permanganate the principal resulting product was veratric acid.

The quantitative composition of the bark oil was about the following: — 12 to 15 per cent. α-pinene; 17 to 20 per cent. camphor; 25 to 27 per cent. safrole and 40 to 45 per cent. methyl eugenol.

In the oil of the leaves, Hargreaves identified: α-pinene (nitrosochloride, m. p. 103°); phellandrene (?); *d*-camphor; a phenol $C_{10}H_{15}O_2$ (?!), and a green compound, boiling at 263°. The quantitative composition was: — 25 per cent. α-pinene and phellandrene (?); 60 per cent. *d*-camphor; 15 per cent. phenols and other compounds.

Strange to say, safrole does not occur in the leaves. Camphor oil affords a similar case, safrole being a component of the wood oil, but not being present in the leaves.

Citronella Oil. When E. J. Parry[1]), a short while ago, advocated the removal of "Schimmel's test" from practice and all professional literature, A. Binz[2]) published a reply, according to which the British objection to this test is easily comprehensible, as this method of commercial analysis has been the means of finding out the British adulterators of the oil coming from Ceylon. It is only since this test is in use that the citronella oil trade has been put on a sound footing. Parry disputes the value and reliability of "Schimmel's test" by stating among other reasons the possibility of finding adulterants of the same solubility as citronella oil, *e. g.* the lower boiling fractions of petroleum. According to his view, the only sure test is an exact chemical investigation, especially the determination of the acetylizable constituents.

Although, as Binz states, these arguments are not at all badly put, they are easily refuted. It is true that the lower boiling fractions of petroleum escape our solubility test; these adulterations, as well as others, can be detected by a more thorough investigation and are found out as soon as the oil gets into the hands of an analyst. The buyer, however, is in most cases not an analyst and can carry out neither acetylations nor other chemical tests. But he is in a position to apply Schimmel's very simple test. It is for this reason that "Schimmel's test" is absolutely indispensable and must be kept at all events. It is the least that can be required of any Ceylon citronella oil and represents so to speak the first coarse sieve, usable for everybody in order to discard bad qualities. The closer investigation by the analyst follows in second line.

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[1]) *Perfum. Record* 7 (1917), 368. — [2]) *Deutsche Parf.-Ztg.* 3 (1917), 57.

The Formosan citronella oil which K. Kafuku[1]) distilled from a grass probably identical with "Indian geranium"[2]), shows the same qualities as the Javanese citronella oil from *Cymbopogon Winterianus,* Jowitt, *viz.,* $d_{17°}$ 0.8868; α_D 24° 24′ (the direction of rotation is not stated); $n_{D17°}$ 1.4700. It contains 84.97 per cent. total geraniol (namely 38.98 per cent. citronellol and 37.23 per cent. geraniol, determined by the phthalic anhydride method) and dissolves in all proportions in 90 per cent. alcohol, in 0.9 vol. of 80 per cent. and in 4.6 vol. of 70 per cent. alcohol.

Clove Oil. According to official communications[3]) the deliveries of cloves in Zanzibar and Pemba for the periods from July 1st, 1914 to June 30th, 1915 and from July 1st, 1915 to March 31st, 1916 were as follows: —

	1914/1915	1915/1916
Zanzibar . . .	6752127 lbs.	4692341 lbs.
Pemba . . .	11598611 „	22101104 „
total:	18350738 lbs.	26793445 lbs.

For the last quarter of this season, 580000 lbs. to 750000 lbs. (?) are still to be expected. The price of the cloves has in no way been influenced by the war. The low level of the prices in the beginning of the year is rather due to the prospective abundant crop than to the state of war.

It appears that at present in Switzerland the market for oil of cloves is overrun with indifferent products, as out of 4 samples we received from that country for examination no less than 3 were objectionable. In each of these 3 cases it was a question of products whose eugenol content had been removed in part, or, what comes to the same, had been mixed with residues from the manufacture of eugenol from oil of cloves. The samples struck at once by their slight smell, and not even one of their constants was able to meet the usual requirements, as is apparent from the comparative data given below. The specific gravities were too low, the rotation figures too high and their solubility and eugenol content unsatisfactory.

	Limits of a good trade product.	Sample I	II	III
$d_{15°}$	1.043 to 1.068	0.9982	0.9988	1.0224
α_D	left, up to — 1° 35′	— 2° 47′	— 2° 56′	— 1° 43′
Eugenol-content, per cent. . .	not below 80	64	65	70
Soluble in 70 per cent. alcohol:	soluble in 1 to 2 vols.	insoluble	insoluble	insoluble.

Even with 80 per cent. alcohol the oils in question only gave clear solutions in concentrations averaging 1 : 1; on further addition of alcohol, the solutions became turbid. The examples cited above may suffice as a warning to be careful in buying oil of cloves.

On the substitution of oil of cloves by methyl benzoate in microscopy see page 91 of this *Report.*

Oil of Cnidium officinale. In Japan the umbellifera, *Cnidium officinale,* Makino occurs whose roots Chinese, Japanese and Corean doctors often employ for various

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[1]) *J. Chem. Ind. Tokyo* **19** (1916), 411; *Journ. Soc. chem. Industry* **35** (1916), 865. — [2]) The usual term for "Indian Geranium oil" is palmarosa oil. — [3]) *Chemist and Druggist* **88** (1916), 953.

nervous disorders and women's diseases and which Sakei[1]) has investigated as to its components. The most important one is a volatile oil, of which it contains 0.82 per cent. It has a brownish-yellow colour, a striking smell and a bitter taste; d 1.030 to 1.040. It is soluble in alcohol, insoluble in water, shows lævorotation and contains an unsaturated acid $C_{12}H_{19}O_3$ (?), an alcohol $C_{10}H_{18}O_3$, and a lactone (?) $C_{12}H_{18}O_2$.

The effect of the oil consists in an increase of the blood pressure by means of stimulating the vaso-constrictors. It also stimulates the central nervous system. It does not attack the kidneys, but apparently produces hæmolysis, when abundantly used.

Elecampane Oil. Concerning the cultivation of elecampane, see page 110.

Essential Oils, Sicilian and Calabrian.

Bergamot Oil. It is easily explicable why just now such volatile oils should be subject to adulteration which only occur rarely in trade or are wholly exhausted. Bergamot oil belongs to this class; the few samples which were submitted to our inspection were nearly all adulterated and we cannot deny ourselves the satisfaction of giving a selection in detail, in order to show partly at least what the quality of this product is now.

A sample from Messina which had been sent us from Switzerland showed the following characteristics: $d_{15°}$ 0.8761; $\alpha_D + 28°$; acid v. 2.7; ester v. 70.0 = 24.5 per cent. ester, calculated as linalyl acetate. The constants prove quite alone that the oil was an inferior, adulterated product. The specific gravity is too low, the degree of rotation much too high and the ester content, which in a good trade sample ought not to be under 34 per cent., quite insufficient. The high rotation indicates an admixture of lemon oil.

Two other specimens were of such quality that one hardly realized if they were adulterated oils or artificial products. One of them showed the following characteristics: $d_{15°}$ 0.8866; $\alpha_D + 15°$; residue on evaporation 6 per cent. (liquid!); acid v. 0.9; ester v. 59.7 = 20.9 per cent. ester calculated as linalyl acetate; soluble in 1 vol. and more of 80 per cent. alcohol. In this case also the much too low ester-content immediately characterized the oil as quite inferior. Strange to say that the oil in spite of its low ester-content dissolves surprisingly well in alcohol, as the majority of bergamot oils do not give a clear solution in 80 per cent. alcohol, and even in the case of the oils that are soluble in it the diluted solution is nearly always turbid; this is all the more to be expected from an oil with a low ester content. Another anomaly finds its expression in the quality of the residue on evaporation which in this case is liquid, whereas it ought really to be of a butter-like consistency. All these facts lead to suspect that it is not the case of a natural oil at all, but that of an unsuccessful artificial product. The same holds good of a second sample which gave the following constants: $d_{15°}$ 0.8864; $\alpha_D + 15° 46'$; acid v. 3.7; ester v. 110.1; saponification number 113.8; acid v. II[2]) 63.5; difference between saponification v. and acid v. II 50.3; residue on evaporation 19.3 per cent.

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[1]) *Mitt. d. med. Gesellschaft zu Tokio* **1916**, 358 (N° 6); *Schweiz. Apotheker Ztg.* **55** (1917), 226. — [2]) Comp. Gildemeister und Hoffmann, *Die ätherischen Öle*, 2nd ed., vol. III, p. 75—77; *Report* October **1910**, 61.

The bad quality of this product was betrayed by the too great difference between the saponification value and acid value II (normally not above 10), as well as by the much too great a residue on evaporation (normally 4.5 to 6 per cent.), which besides apparently consisted only of a fatty oil and probably contained none of those wax-like components which are so characteristic for bergamot oil. As fatty oils have a high saponification value, it has evidently been added with the object of leading to assume the presence of a bergamot oil rich in linalyl acetate, as the above-mentioned ester value would lead to the assumption of about 38.5 per cent. linalyl acetate. In reality, however, this content is considerably smaller and can approximately be calculated from the acid value II, which would correspond to a linalyl acetate quantity of 22.2 per cent. As indicated above, this oil also gives decidedly the impression of an artificial product whose manufacture has been a complete failure.

A sample handed to us for investigation by a Leipzig firm, with the proud title *"Oleum Bergamottæ Ia Reggio rect. pur."*, was simply an artificial, about 80 per cent. linalyl acetate, as its constants prove: $d_{15°}$ 0.8997; α_D — 10° 11′; $n_{D20°}$ 1.45082; acid v. 1.9; ester v. 233.3, corresponding to 81.7 per cent. linalyl acetate, soluble in 2.7 vol. and more of 70 per cent. alcohol.

Sweet Orange Oil. A sweet orange oil which recently came to hand, is also worth mentioning at this point, to serve as a warning. It was quite sufficient to determine the specific gravity and optical qualities of it, in order to form an opinion on its quality. The constants indicated were as follows: $d_{15°}$ 0.8662; $\alpha_{D25°}$ + 58° 18′; α_D of the first 10 per cent. of the distillate + 56° 20′. Compare with this the characteristics of genuine sweet orange oil, namely, at 15° a specific gravity of 0.848 to 0.853 and an optical rotation of + 95° 30′ to + 98°; the first 10 per cent. of the oil having a rotation not measurably less than the original oil.

The extreme divergence from the normal figures makes it quite obvious that the sample is at least greatly adulterated. Probably, however, this is an artificial product in which lemon oil terpenes have been employed, whereas orange oil is entirely absent, which can be inferred, above all things, by the exceedingly low degree of rotation of the first 10 per cent. of the distillate (boiling above 172°). However that may be, there is at least no doubt that this is again a case of a most vulgar fraud.

G. C. Hood[1]) publishes an account of the oil content of Florida oranges. At various periods he collected the oranges during harvest time and made a determination of the oil value of the peel. Thereby the fact came to light that conditions of climate and culture greatly influence the oil content of the fruit which besides varies considerably with the species. Only when fully ripe does the peel contain its highest percentage of oil, it is, however, sufficiently great to permit of collecting even in unripe fruit which cannot be gathered. Rain during harvest reduces the oil yield considerably. The occurrence of a rust mite has no effect on the oil yield of the ripe fruit. In tabulated form Hood reports on the weekly oil yield for 8 orange varieties from 17 different localities in Florida.

As to the export of lemons and oranges from Syria, see p. 107.

[1]) *Journ. ind. eng. Chemistry* 8 (1916), 709; *Perfum. Record* 7 (1916), 310.

Eucalyptus Oil. There were, of course, absolutely no imports of Australian eucalyptus oils, but fortunately, we were able to put at the disposal of the consumers large quantities of synthetic Globulus oil, a product which, in addition to a high content of eucalyptol, has all the qualities of the genuine distillate and may, in consequence, be used as a substitute of this without any hesitation. We shall most likely be in a position to continue manufacturing this welcome makeshift in sufficient quantities.

A curious sort of eucalyptus oil was offered to us in the course of the summer. A considerable quantity was supplied to us at the beginning of the war by a Hamburg firm as Globulus oil, but was only utilized in part by us, so that a largish portion remained over. Its strong smell of camphor oil immediately characterized this product as a gross imposition, which fact was also verified by investigation, as its qualities differed entirely from those of Globulus oil, *viz.*: $d_{15°}$ 0.8865; $\alpha_D + 21° 15'$; insoluble in 70 per cent. alcohol. In the case of oil from *Eucalyptus Globulus* the figures show the following limits: $d_{15°}$ 0.910 to 0.930; α_D dextrorotatory up to $+ 15°$; soluble in 2 to 3 vols. and more of 70 per cent. alcohol. Moreover, the percentage of eucalyptol in the sample was quite insufficient, since it amounted but to about 30 per cent., whereas a good Globulus oil contains 80 per cent. or more.

It is not the question of an oil of another eucalyptus species, on the contrary, it is evident from the above-mentioned overpowering smell of camphor oil, in conjunction with its other qualities, that the product consisted of a cineoley camphor oil fraction which to the utmost may have been scented with eucalyptus oil. The Hamburg firm, mentioned above, evidently imagines that for medical purposes a product may be indifferently bad in quality. A point of view which is probably highly remunerative, but implies a doubtful kind of patriotism.

On page 56 of our October *Report* for **1913** we mentioned that R. T. Baker and H. G. Smith came to the conclusion, as the result of their investigation of Tasmanian eucalyptus species, that the *Eucalyptus amygdalina* which grows there is not identical with the continental *amygdalina species* which they termed *E. a.* var. *Australiana*, although by rights it ought to be given specific rank from *E. a.* A rectification in this concern was published by the authors in their last paper on *Eucalyptus Australiana, spec. nov.* ("Narrow-leaved Peppermint") and its volatile oil[1]). The new species grows on the ranges of New South Wales and Victoria, in a region which has little agricultural value; as this species produces a large quantity of good oil, they propose to establish a permanent industry.

One easily obtains a good product rich in cineole on collecting the steam-distillate during the first hour separately. It contains more than 70 per cent. cineole, whereas the amount of cineole in the total product is only 45 per cent. Besides, the first fraction is nearly colourless and contains mere traces of aldehydes, so that it is sufficiently pure for pharmaceutical purposes, whilst the yield is as great as that of the total oil in most of the other eucalypts. The portions of the oil holding phellandrene which pass over particularly in the later distillates, appear to contain an alcohol $C_{10}H_{18}O$.

Oil of Euthamia Caroliniana. *Euthamia (Solidago) Caroliniana*, (L.) Greene is a composite occurring in a part of the southern U. S. and found most frequently on the coast, especially of Florida. G. A. Russell[2]) has investigated the oil distilled from

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[1]) *Journ. and Proceed. Royal Soc. of N. S. W.* **49** (1915), 514; *Journ. chem. Soc.* **110** (1916), I. 566. — [2]) *Journ. Americ. chem. Soc.* **38** (1916), 1398.

the fresh herb which had been gathered near Orlando (Orange county, Fla.) before blossoming. Yield, 0.693 per cent. It has a light yellow colour and a pleasant, aromatic odour. Constants: $d_{23°}$ 0.8587; α_D — 10° 48′; n_D 1.4805; ester v. 6.35; ester v. after acet. 25.3; easily soluble in 6 vol. of 90 per cent. alcohol; slightly turbid when dissolved in 9 vol. 70 per cent. alcohol and more. The oil contained no phenols, but evidently aldehydes (reaction with magenta-sulphurous acid). The main component is dipentene which was identified by its tetrabromide melting at 124°; possibly the oil also contains pinene, limonene, and esterified formic and acetic acids.

Fennel Oil. We are without any news as to whether, in consequence of the Russian invasion of the Bukowina and East Galicia, it will be possible to take up fennel growing again in this district in the near future, as the distillers and buying agents of the Skala district have been able to return only quite recently. Fennel oil is not to be obtained anywhere, and the fennel seeds offered now and then from the Lützen district were far too expensive to be distilled. He who formerly employed fennel oil must use anethole at present, which is still to be had in large quantities and at acceptable prices.

Geranium Oil. A sample of geranium oil submitted to us for examination may also be mentioned at this point, in order to prove to our readers in what a villainous fashion people are defrauded nowadays. The oil gave the following constants: $d_{15°}$ 0.9573; α_D + 1°; acid v. 12.0; ester v. 7.5; soluble in 1.5 vols. of 70 per cent. alcohol and showing opalescence in a dilution beyond about 6 vols. Quite apart from the fact that the constants differ in a greater or lesser degree from normal ones, the smell alone testified that the product had nothing whatever to do with geranium oil. It was a resinous oil containing *iso*pulegol, apparently consisting of residues, such as are left over in the manufacture of citronella oil, and which at best was perfumed with a little geranium oil. The prospective buyer was evidently quite right in consulting us in advance about the quality of the oil, as he thereby saved himself both loss and annoyance.

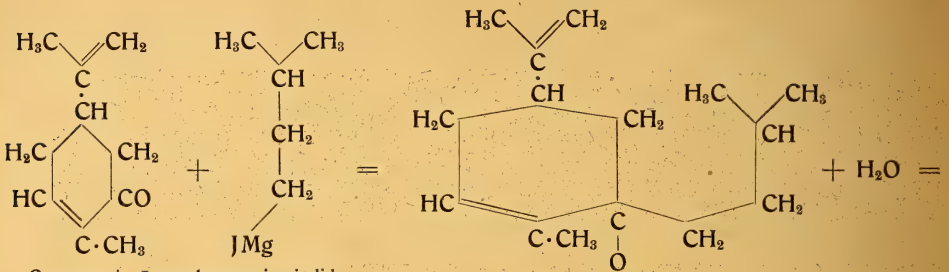
Oil of Gum Ammoniac. The greater part of this substance which finds its way into European trade comes from Persia and is extracted from the Umbellifera *Dorema Ammoniacum*, Don., whereas *Ferula communis* var. *brevifolia*, Mariz. provides the Moroccan and *F. marmarica*, Aschers. et Taub. the Cyrenian drug. Nothing was hitherto known about the composition of this oil and it is only recently that P. Roenisch[1]) published some particulars about it. The commercial oil (which we supplied) showed the following properties: $d_{20°}$ 0.8855; α_D + 1° 42′; n_D 1.47233. A combustion gave a yield of about 81 per cent. carbon and 12 per cent. hydrogen which indicated that the oil contained considerable quantities of substances rich in oxygen. Nearly all of the already known components of volatile oils contain less hydrogen, which led Roenisch to the conclusion that the oil contained hydrogenized sesquiterpene compounds, a supposition which proved to be right.

The oil boiled between 60 and 190° (10 mm.). The analyses of the fractions gave higher figures for hydrogen the more the boiling point rose. Accordingly Roenisch

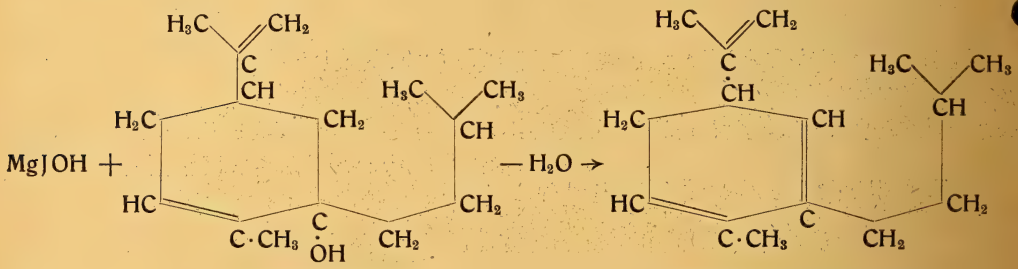
[1]) Investigation of oil of gum ammoniac and synthetic experiments with a view to elucidate its components. Inaug.-Dissert., Breslau 1916.

suspected the presence of olefinic and monocyclic sesquiterpene compounds in the fractions with a higher boiling point, chemical compounds which have hitherto been comparatively little examined. Oxygenated derivatives of the monocyclic sesquiterpene series have only been described quite recently, so that the author found it necessary to produce such compounds by synthetic means, to enable him by comparing their properties with those compounds found in the oil of gum ammoniac to draw conclusions as to the composition of the latter.

Semmler and Jonas[1]) carried out the first syntheses in the sesquiterpene series. For instance they obtained *cyclo*isoprenemyrcene by heating myrcene and isoprene in a sealed tube. This mode of procedure, however, by no means excludes the possibility of molecular rearrangement; whereas Grignard's[2]) reaction supplies a quite reliable



Carvone + *Iso*amylmagnesiumiodide.



Compound $C_{15}H_{26}O$. *Iso*amyl-α-dehydrophellandrene.

method for this synthesis. By the action of *iso*amylmagnesiumiodide on *d*-carvone in a solution of benzene, Roenisch obtained a compound $C_{15}H_{26}O$ which immediately split off water and produced a sesquiterpene $C_{15}H_{24}$, boiling between 130 and 132° (11 mm.) and showing the following properties: $d_{22°}$ 0.8679; α_D + 18° 30′; $n_{D20°}$ 1.49478. These figures are quite in accord with the properties of the hitherto known monocyclic sesquiterpenes, bisabolene (limene), zingiberene, and *cyclo*isoprenemyrcene. Theory demands that the synthetic hydrocarbon should contain a pair of conjugated double bonds, besides that it contains a third double bond in the *iso*propylene group; accordingly it can be characterized as an *iso*amyl-α-dehydrophellandrene, a dehydrated α-phellandrene, in which the *iso*amyl group takes the place of one hydrogen atom.

Phellandrene with conjugated double bonds in the nucleus can by no means be reduced by the action of sodium and ethyl alcohol, on the contrary with facility by

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[1]) *Berl. Berichte* **46** (1913), 1569; **47** (1914), 2080; *Report* October **1913**, 35; April **1915**, 85. — [2]) Comp. M. Bruhnke, Inaug.-Dissert., Breslau 1915.

the combined action of sodium and amyl alcohol. Nor do sodium and ethyl alcohol reduce *iso*amyl-α-dehydrophellandrene, whereas the use of amyl alcohol effects at least a partial reduction. The chemical proof of the presence of the 3 double bonds in the synthetic sesquiterpene was supplied by the hydrogenation by means of hydrogen in the presence of platinum black. This led to the formation of a hydrocarbon, $C_{15}H_{30}$, tetrahydro*iso*amyl-α-phellandrene, whose boiling point lay between 131 and 133° (14 mm.) and which gave the following constants: $d_{22°}$ 0.8250; α_D — 1° 30′; $n_{D20°}$ 1.45562. Its properties are quite in accord with those of hexahydrobisabolene, hexahydrozingiberene, and of hexahydro*cyclo*isoprenemyrcene[1]). The action of hydrochloric acid on the synthetic sesquiterpene does not lead to the formation of a solid addition-product.

Besides the above-mentioned sesquiterpene, Roenisch obtained a small quantity of a higher-boiling fraction which apparently contained the alcohol $C_{15}H_{26}O$ belonging to the sesquiterpene. This fraction was further investigated by Semmler and Oelsner[2]). Whereas Roenisch had made his investigations in an absolute benzene solution with the addition of only a small quantity of absolute ether, Semmler and Oelsner succeeded in obtaining a better yield of products containing oxygen by working in an absolute ethereal solution. Strange to say they obtained by this means chiefly a sesquiterpene ketone, $C_{15}H_{26}O$. Under these conditions Grignard's reaction had taken an abnormal course by the addition of the amyl radicle to the double bond, instead of to the oxygenated carbon atom. This ketone, *iso*amyldihydrocarvone, had a boiling point between 144 and 149° (10 mm.) and the following constants: $d_{20°}$ 0.9022; α_D — 8°; $n_{D20°}$ 1.47694. Reducing agents, such as sodium and alcohol, produced a monocyclic alcohol, $C_{15}H_{28}O$ (boiling point 150 to 155° [10 mm.]; $d_{20°}$ 0.8993; α_D — 1°; $n_{D20°}$ 1.47449). The hydrogenation of this alcohol with only one double bond with the help of platinum and hydrogen produced the saturated monocyclic alcohol $C_{15}H_{30}O$; boiling point 145 to 152° (12 mm.); $d_{20°}$ 0.8906; α_D — 1°; $n_{D20°}$ 1.46855.

Besides the monocyclic ketone, Semmler and Oelsner obtained a bad yield of a tertiary alcohol, $C_{15}H_{26}O$, *iso*amyldihydrocarveol; boiling point 155 to 170° (12 mm.); $d_{20°}$ 0.9217; α_D — 4°; $n_{D20°}$ 1.4917. A natural monocyclic sesquiterpene alcohol, $C_{15}H_{26}O$, elemol, had already been isolated from elemi oil by Semmler and Liao[3]).

By means of the synthetic experiments mentioned above, Roenisch had proved that monocyclic sesquiterpene products containing oxygen were not to be found in ammoniacum oil, as they all showed a much higher specific gravity than the fractions of this oil. As the possibility still remained that products of the aliphatic series were present in the oil, he hydrogenized with the help of hydrogen and platinum the aliphatic sesquiterpene alcohol, farnesol, in order to obtain comparative data, and obtained hexahydrofarnesol; boiling point 145 to 155° (15 mm.); $d_{22°}$ 0.8387; $\alpha_D \pm 0$; $n_{D22°}$ 1.44525. However, he did not succeed in producing the simply and doubly unsaturated alcohol whose properties might have served for the identification of the compounds found in gum ammoniac oil.

It is highly probable that in the lower-boiling fractions of the gum ammoniac oil a terpene was present which, however, it was quite impossible to identify. The saponified fractions, boiling between 68 and 120° (11 mm.), contained *d*-linalool. It was purified over the sodium compound and characterized by its phenylurethane (melting point 65°). Apparently, the linalool is present as acetate. Citronellyl acetate also is, as it seems, present in the lowest-boiling fractions. Oxidation by means of bichromate of

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[1]) Comp. *Report* October **1913**, 34, 136; April **1915**, 85. — [2]) Unpublished paper. — [3]) *Berl. Berichte* **49** (1916), 796; *Report* October **1916**, 20.

potash and sulphuric acid apparently transformed the terpene alcohols into a mixture of citral and citronellal. Even prolonged boiling over sodium did not make the fractions boiling between 125 and 135° (12 mm.) free from oxygen owing to the presence of a sesquiterpene ketone $C_{15}H_{26}O$. They contained a hydrogenated monocyclic sesquiterpene $C_{15}H_{26}$, ferulene. In order to remove the ketone, Roenisch reduced the fractions with sodium and alcohol, whereby the ketone turned into the alcohol $C_{15}H_{28}O$ which he then destroyed by boiling with sodium. The purified hydrocarbon thus obtained gave the following constants: boiling point 126 to 128° (10 mm.); $d_{20°}$ 0.8687; $\alpha_D + 6°$; $n_{D20°}$ 1.48377. Ferulene was also freed from the ketone by other means, namely, by treatment with hydroxylamine. The ferulene thus produced gave the following constants: boiling point 124 to 126° (7 mm.); $d_{20°}$ 0.8698; $\alpha_D + 6°$; $n_{D20°}$ 1.48423. These lead to the conclusion that it is not quite pure. It contained two double bonds which could be hydrogenated by means of platinum and hydrogen, thereby forming tetrahydroferulene which boiled between 118 and 122° (10 mm.) and gave the following constants: $d_{20°}$ 0.8400; $\alpha_D + 4.2°$; $n_{D20°}$ 1.45810.

The aliphatic ketone $C_{15}H_{26}O$, doremone, also occurs in the fraction boiling between 140 and 155° (10 mm.); after purification by means of repeated distillation it gave the following constants: boiling point 145 to 155° (12 mm.); $d_{20°}$ 0.8765; $\alpha_D + 3° 30'$; $n_{D20°}$ 1.47160. The oxime melts at 88°, the semicarbazone at 124°. The ketone could not be recovered from the oxime. Sodium and alcohol as reducing agents transformed the doremone into doremol which boiled between 145 and 150° (12 mm.) and gave the following constants: $d_{20°}$ 0.8702; $\alpha_D + 3°$; $n_{D20°}$ 1.47130. On hydrogenation by means of platinum and hydrogen; this produced the saturated alcohol, $C_{15}H_{32}O$, tetrahydrodoremol, boiling between 140 to 145° (12 mm.), and showing the following constants: $d_{20°}$ 0.8403; $\alpha_D + 2° 48'$; $n_{D20°}$ 1.44817. When reduced by means of platinum and hydrogen doremone is transformed into tetrahydrodoremone which boils between 142 and 144° (12 mm.) and gives the following constants: $d_{20°}$ 0.8434; $\alpha_D + 1° 24'$; $n_{D20°}$ 1.44803. Doremone is the first aliphatic sesquiterpene ketone found in a natural state.

On being treated with acetic anhydride and sodium acetate, doremoneoxime is transformed into an acetate, which leads to the conclusion that a ketoxime is present, as aldoximes produce nitriles under these circumstances.

Roenisch obtained by means of repeated fractional distillation of the higher-boiling compounds of ammoniacum oil an acetate of an aliphatic sesquiterpene alcohol, doremylacetate, which showed the following constants: boiling point: 155 to 165° (12 mm.); $d_{20°}$ 0.8896; $\alpha_D + 4° 48'$; $n_{D20°}$ 1.46596. The free alcohol, doremol, boiled between 145 and 155° (12 mm.); $d_{20°}$ 0.8723; $\alpha_D + 3° 30'$; $n_{D20°}$ 1.47216. It appears to be identical with the doremol obtained by reducing doremone, as platinum and hydrogen first produced dihydrodoremol boiling between 140 and 150° (12 mm.) and giving the following constants: $d_{20°}$ 0.8584; $\alpha_D + 3°$; $n_{D20°}$ 1.45916, this being subsequently transformed into tetrahydrodoremol.

The fraction of the gum ammoniac oil boiling above 175° (12 mm.) became, on cooling, a solid mass resembling paraffin, which contained a paraffin alcohol, $C_{16}H_{34}O$, melting at 48°, the latter being highly probably identical with cetyl alcohol. Hitherto this had only been found in the animal organism.

Hyssop Oil. We were in a position to distil fairly large quantities of hyssop of our own growing and shall be able to extend our fields still further after the war. We may hope then to succeed in eliminating little by little all imports of French oil, which, by the way, does not come up to ours.

Oil of Kæmpferia Ethelæ. This oil, occurring in the tubers of *Sherungulu,* which E. Goulding and O. D. Roberts[1]) have thoroughly investigated, has recently been distilled again in the London Imperial Institute[2]). The yield amounted to 0.45 to 0.55 per cent., expressed on the moist material, and 1.80 to 2.35 per cent., expressed on the tubers dried at 100° C. The oil showed the following constants: $d_{15°}$ 0.924; $\alpha_D + 26° 42'$; acid v. 1.0; ester v. 11.5; ester v. after acet. 33.6. The smell is unpleasant and is not appreciated by perfumers; at best the oil might be used as a substitute for oil of spike lavender, hence it would not be worth while to produce it. A further disadvantage it shows is that the higher-boiling fractions of the oil have only a slight odour, hence it lacks persistance. The tubers, on being freshly dug up, smell first of all of linalool and of methyl anthranilate, the cineole odour only being noticed after cutting the tuber.

Lavender Oil. In the *Dokumente zu Englands Handelskrieg,* edited by A. Hesse and H. Grossmann[3]), an article has been published on lavender oil at the Lyons and Leipzig fairs, which we here publish in full, with regard to the great interest it has for our branch of industry. It runs as follows: —

"At Mr. Barlatier's lecture, on Sunday, July 23rd, 1916, at Lyons, Mr. Herriot, the mayor of our city and senator of the Rhône department, judiciously explained that it would not suffice to put heavy duties on the goods of the *boches,* after the war, in order to protect the French market against being flooded by them, but that our country must make a decided start already now to manufacture those goods, which, out of thoughtlessness, or, let us say it boldly, out of laziness, it was used to buy from our enemies on the farther side of the Rhine.

Mr. Herriot laid special stress on dyes, the wholesale suppliers of which the *boches* had become for France and which we must, in the future, manufacture at all events in our own country. The same ought to be the case with floral oils.

In the number of June 18th last, *Le Reveil National* published, under the heading: "An Industry to be developed in France", an article on the cultivation of the rose for obtaining otto of roses, in order to free the French perfumery industry of the enormous tribute, paid before the war to our old and false friends, the Bulgarians, from whom, year after year, otto of roses was bought at tremendons prices. In 1909, the Bulgarian production amounted to 4316 kilos, 2336 kilos of which, *i. e.* more than 54 per cent., were exported to France. Calculating the value at the last price, which was 3500 Francs, it comes up to the considerable sum of 8.17 million Francs.

But there is another essential oil, the manufacture of which the *boches* have tried to monopolize, *viz.,* oil of lavender. The firm of Schimmel in Leipzig erected for this purpose, a few years ago, two very considerable factories: one at Barrême (Basses-Alpes), the other at Sault (Vaucluse). Schimmel there distilled lavender and spike flowers from the mountain districts of the Alps and the Mont Ventoux, where the harvest is in full swing at present.

Both factories are sequestered and it would seem as if the small distillers of the region (150 in the Department of the *Basses-Alpes* alone) could now use the flowers. Unfortunately, great numbers of them are at the front, apart from those that have been killed in action, wounded, or made prisoners. On the other hand, there is a scarcity of women for picking the flowers, as they are mostly

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[1]) *Journ. chem. Soc.* **107** (1915), 314; *Report* October **1915**, 24. — [2]) *Bull. Imp. Inst.* **14** (1916), 378. — [3]) *Chem. Industrie* **40** (1917), supplement to N° 24, p. 1120. — *Le Réveil National* (Lyons) Aug. 8th, 1916.

occupied with other harvest work, so as not to endanger the nation's food supply.

In order to take advantage of the lavender and spike crops at the right moment, it is desirable that the industry of Lyons, which means to compete with the famous Leipzig fair, would use its capitals to foster the distillation of these flowers in the Alps, which the *boches* tried to lay hands upon before the war, in order to beat the Grasse perfumers on the world's market. We called the attention of the Lyons Chamber of Commerce to this matter and to the advantage which it would bring to attract the oil producers of the whole South East of France to the next Lyons fair.

Although nothing has been done as yet in this direction, it is never too late to mend. It has been reported already that Mr. Herriot, our venerated senator and mayor, intends to stay in the *Basses-Alpes* on one of his next journeys, in order to canvass, by personal influence, exhibitors for the Lyons fair of 1917. It is to be hoped that, for the sake of competing with Leipzig, he will try the seemingly impossible in order to transfer the trade with perfumes and aromatics to his town".

The editors add the following postscript: "In French journals of late years, a tremendous number of articles have appeared which, though small, were often cleverly written and had the aim of making a keen and skilful propaganda for the extension of the native chemical industry. We have, so far, published only a small selection of these articles, for, in the attempt to collect them all one would have wasted almost entirely the space reserved for better productions. Nevertheless, these newspaper reports, which are collected in other quarters[1]), show how France has recognized the faults of its chemical industry and now attempts in all possible ways to repair them. Any means will do, from the justified criticism of the sometimes really incomprehensible conditions, to lies and deceit. A good example of the contortion and exaggeration of existing conditions is above article, which we, therefore, do not want to keep from our readers, especially as it has been further criticized hereafter by a well-informed authority. We would mention here one subject of the article that has not been taken into consideration in said criticism; *viz.*, the remarks on France's dependence on the Bulgarian rose industry. Everybody acquainted with the conditions knows that this dependence is universal and by no means limited to France, whose rose fields in the neighbourhood of Grasse scarcely suffice to supply the flowers required for rose pomades and extracts. The distillation of otto of roses in the South of France is quite inconsiderable and will never be able to compete with the vast rose growing regions in Bulgaria. The calculation, made in above article on the basis of a price which has been reached only now under quite exceptional circumstances, likewise shows that any means will be deemed good if it helps to attain the purpose in view".

Thereafter, an article is reproduced which appeared in the *Deutsche Parfümerie-Zeitung* (2 [1916], 333) under the title of *"L'essence de Lavande aux Foires de Lyon et de Leipzig"*. It runs as follows: —

Under above heading and with the sub-title *"la maison Schimmel de Leipzig accaparait notre Lavande, la foire de Lyon la propagera dans le monde entier"*, M. Galfard, *ancien conseiller du commerce extérieur de la France*, published on

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[1]) Comp. E. Oberfohren, *Französische Bestrebungen zu Verdrängungen des deutschen Handels. Kriegswissenschaftliche Untersuchungen aus dem Institut für Seeverkehr und Weltwirtschaft a. d. Univ. Kiel.* Jena 1916.

August 6th, 1916, in *Le Réveil National* at Lyons, an article which cannot remain unchallenged. It is for the sake of truth, which virtue seems to have died years ago in that part of the world now hostile to us, especially in the so-called chivalrous France, that some of the very coarsest lies and distortions of said article are here impressed upon the public.

The heading gives us to understand, and the article enlarges upon the idea, that lavender oil is sold at the Leipzig fair and that the coming Lyons fair will bring about a change in this direction. The author knows, no doubt, in how far he may trust the ignorance of his readers, else he would not dare to build up the whole article on a fiction, for: lavender oil is not offered and sold at all at the Leipzig fair.

Another untruth, which the author uses as a weapon against German industry, is the assertion that German enterprises tried to monopolize lavender oil and to get hold of the French lavender crop: *"ces fleurs des Alpes, dont les Boches cherchaient avant la guerre à accaparer les essences, afin de battre les parfumeurs de Grasse sur le marché mondial"*. The writer thus, quite unconsciously, lays himself and his countrymen open to ridicule and, besides, the matter is quite different. The firm of Schimmel & Co., Miltitz near Leipzig, which Mr. Galfard focusses quite specially in his chauvinistic tirades, was compelled by dire necessity, not induced by inclination, to distil lavender oil in the South of France, having found out by the experience of several decades that the Grasse factories were quite unable to supply it with the required quantities of pure oil. The firm's analytical records give clear evidence as to date, name of the suppliers, and all other details, showing why Schimmel & Co. finally had to arrive at the decision to start distilling lavender oil in the producing districts ..... simply because the adulterations, practiced systematically by the Grasse people, had taken such proportions as to make it more and more difficult to get pure lavender oil at all. It is superfluous to mention that Schimmel & Co. did not make a fortune there, a fact proved beyond doubt by the relative book-keeping. From the beginning, however, the firm met with a systematic press campaign of all the French provincial papers. At first it was said that the Germans pushed the prices, in order to get flowers and oil in their own hands. In consequence, the Grasse people offered ever increasing prices in order to cut off the supplies of the new undertaking. As then Schimmel & Co. withdrew from the flower market and put up ambulant distilleries in order to obtain in this way part of the oil required, leaving the flower dealers aside, so as to induce them to make more reasonable prices, the sensational press went into another rage. There it was to be seen, it was said, how certain people had installed themselves in the lavender districts in order to worry the poor peasants by cutting the prices to such an extent as to make it unprofitable to gather the flowers. On the world's markets, the rumour was spread, one year, that Schimmel & Co. pushed the prices, Schimmel & Co. were responsible for the bull movement. The next year, Schimmel & Co. were the cause of the bear tendency, and so forth; in short: whatsoever Schimmel & Co. might do or omit, the journalists of the chivalrous nation always met them with misrepresentations or suppression of the true state of affairs, never with truth, their motives being envy and chauvinism.

And withal the whole management of Schimmel's branch establishments was in French hands up to the last day of peace. There was no other desire but to produce the quantities of lavender oil needed by the firm in its own business. *Hinc illæ lacrimæ!*

Another, simply appalling lie, which was propagated in addition, some years ago ..... the honourable "knight" in question called himself Marcel Provence ..... was, that Schimmel's factory at Barrême was built and kept in working order with the only aim and object of spyism. As it was within so and so many yards off the railway line, no special strategist was required to find out what this meant, and so forth. One even "discovered" that a harmless gable ornament, put up, as it was, by a French bricklayer on his own accord, represented the hated spiked helmet, which had evidently been placed there as the symbol of Prussianism, and one publically proclaimed oneself as saviour of the country on account of having found out and unmasked this danger in time!! The common sense of the French people must have come to a bad pass, if a journalist of the third or fourth class may venture to put such nonsense before his readers without finding any opposition. But everything seems to be possible in France, as long as it concerns Germany and German firms. Then no nonsense is too nonsensical to be offered to the public at once; the public believes everything, if it is only something spitefully hostile towards Germany.

It was reserved for Schimmel's factories to show what pure lavender oil is and ought to be, if it is distilled in the proper way. Before, the world had to be satisfied with what Grasse firms chose to put before them as "pure" oil of lavender. A glance at the French price-lists with their one or two dozens of essences de Lavande *"Mont-Blanc"*, *"extra"*, *"fleurs mondées"*, *"extrafine"*, *"surfine"*, *"fine"*, *"supérieure"*, &c., &c. must give food for thought to the reasoning mind! But the French, who formerly were capable of thinking logically especially in scientific and chemical matters, went so far as to assert that Schimmel's product was made up, the best proof of it being that nobody in the South of France had ever succeeded in distilling oils of so high a grade. In short ..... the articles were all of this tenor for years ..... *ad nauseam* ..... and the less proof of their veracity could be given, the greater the clatter, the more sullen the intrigues and the systematical mischief-making".

P. Maure[1]) has been able to record especially good results with the cultivation of lavender in the South of France. The peasants of the Basse-Provence had planted their lavender plants too closely together in the hope of realizing greater profits by this means. The average space between the plants only amounted to 50 and 60 cm., and the consequence was that the plants in a short time were so cramped that light and air were totally excluded. Maure accordingly recommended an intervening space of at least a yard and a half. In 1914, plantations were therefore started at a height of 340 m. on ground facing north as well as south. A southerly field was stocked with plants of *Lavandula delphinensis* and *fragrans* which had been collected in the Alpes du Divis at a height of 1200 m. The field was manured with artificial fertilizers, hoed, and kept free from weeds. In 1916, this lavender produced an oil of the specific gravity of 0.89029 (15°) and of an ester content equal to 44.59 per cent. Lavender from plants on ground facing north contained 41.16 per cent. linalyl acetate and showed d_{150} 0.89176. 100 kg. of lavender blossoms produced 1.06 kg. of oil = 1.06 per cent.

In 1913, two plots in a garden had been planted, one with *Lavandula delphinensis*, the other with *L. fragrans*. Owing to the dry summer of 1916 the plantations were watered in such a manner that the lower part of the plants was nearly continually

[1]) *Parfum. moderne* **9** (1916), 121.

covered with water. In spite of this unceasing moisture the quality of the oil had not been affected. *Lavandula delphinensis* produced an oil containing 48.02 per cent. linalyl acetate ($d_{15°}$ 0.89004), and *L. fragrans* an oil with 47.53 per cent. linalyl acetate ($d_{15°}$ 0.88606).

The Dutch Pharmacopœia prescribes for the production of spirit of lavender to macerate lavender blossoms with alcohol and to distil the latter after 24 hours. According to the German Pharmacopœia, 5th ed., lavender blossoms are mixed with alcohol and left in a closed vessel which is repeatedly shaken during 24 hours, after which process 4 parts are distilled off from the mixture. Both prescriptions refer of course to the use of dry blossoms. According to P. van der Wielen[1]), one obtains the best preparation by distilling fresh blossoms with spirits of wine; one can calculate on the average that 100 g. of fresh blossoms correspond to 25 g. of dry ones.

The genus *Phoma* belonging to the class of the *Sphæropsidales* or *Pycnide* fungi includes more than 1100 species which have been observed on all possible nutritive plants. A whole number of species produce plant diseases, among them *Phoma lavandulæ*, Gabotto. This species had not been found hitherto in England, but according to W. B. Brierley[2]) it became rather pernicious there in the summer of 1916. The author had observed two lavender beds on which nearly all the plants died. The twigs affected by the disease had a dry appearance and acquired a dirty brownish-grey colour; the epidermis broke up into little silver-grey scales. The leaves retained their normal appearance for some time, then faded pretty fast, became brown and shrivelled up. The parts of the plants, situated above the dry, discoloured spots, decayed. The disease spreads quickly and frequently produces great damage. On examining a dead twig, it appeared that the shrivelled-up part under the scaling-off epidermis was interspersed with little black dots, the pycnides of the fungus mentioned above. In order to prevent the spread of the disease, all infected twigs must be removed, if possible even before the pycnides have been formed.

Lemongrass Oil. There are two kinds of lemongrass oil, the easily soluble East Indian from *Cymbopogon flexuosus*, Stapf, and the sparingly soluble, so-called West Indian, from *C. citratus*, Stapf. S. C. Hood[3]), in a noteworthy treatise on the possibility of producing lemongrass oil in the U. S., leaves the question undecided whether there really are two species, but supposes that there are numerous local varieties. The Bureau of Plant Industry of the U. S. made experiments of cultivation with 13 varieties from eight different places of origin. The best results were obtained on well-drained, sandy clay, but the plant likewise grows well on light sandy soil, as is to be found in the high pine districts of Florida. The results were also good on the freshly prepared sandy soil of pine woods, without any addition of lime. Badly drained soil and soil with rocks at the bottom at a depth of about 3 feet ought not to be planted with lemongrass. No experiments have been made on heavy clayey soil, but it is to be presumed that it is not appropriate for growing lemongrass.

The lemongrass plant requires subtropical climate. Where the temperature never drops below — 4°, one may plant lemongrass without any fear, for it stands an even somewhat lower temperature.

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[1]) *Pharm. Weekblad* **53** (1916), 872. — [2]) *Kew Bull.* **1916**, 113; *Pharmaceutical Journ.* **97** (1916), 85. — [3]) *Bureau of Plant Industry*, U. S. Dep. of Agriculture, Bull. 442; *Americ. Journ. Pharm.* **89** (1917), 180.

In Florida, the grass does not run into seed, only sometimes a stunted inflorescence will form on old neglected plants; propagation takes place by division. A full-grown plant will furnish twenty-five to fifty new plants, which must be cut down to about 3 inches before being planted. This is done early in spring, if possible after rain, in rows 3 feet apart, the distance between the plants in the rows being 18 inches.

The results of manuring experiments indicate that, on sandy soil, lemongrass requires more potash than most of the other grasses. The analysis of the different varieties showed great differences in the contents of nitrogen, phosphoric acid, and potash. 5 tons of one variety contained 20.32 lbs. of nitrogen, 33.20 lbs. of potash and 18.75 lbs. of phosphoric acid. For manuring purposes, it is best to use potash in the form of the sulphate and nitrogen must be added in organic form. The best results were obtained with a manure containing 4 per cent. of nitrogen, 5 per cent. of potash and 8 per cent. of phosphoric acid, and of which 600 lbs. were used per acre. It is necessary to loosen the soil frequently in spring and to weed carefully in summer, for a trifling admixture of badly smelling weeds may spoil the smell of the oil.

The first cut takes place four or five months after planting, when the plants ought to be 2 1/2 to 3 feet high and 8 to 10 inches apart. They may be cut once more early in autumn. There may be three crops in the second year. Mowing machines may be used for cutting if they are adaptable, so as to cut the grass about 8 inches above the soil. It is not necessary to cut the stalks too low down, as the lower parts contain but little oil, although rich in citral.

Drying causes only a small loss of oil, the yield being 0.37 per cent. from fresh grass, 0.31 per cent. from grass dried in sunshine and 0.32 per cent. from artificially dried grass, calculated with regard to fresh grass; the citral contents were 77.78 and 79 per cent., respectively, according to the sulphite method. As shown, the proportion of citral remains unaltered, but the solubility of the oil from dried grass decreased more rapidly on storage than that of the oil from fresh material.

The usual stills are taken for distillation. By cutting the grass, one succeeds in getting larger quantities into the stills, but this in no way facilitates the distillation. The yield may be increased by using steam of about 10 atmospheres of pressure, but the oil thus obtained is darker and its citral content lower. The water used in distilling contains so little oil (1.2 gram per gallon) that it would not pay trying to obtain it.

Several kinds of lemongrass have been cultivated, belonging to the West Indian, the East Indian, and the Mexican types.

The yield as well as the citral content are strongly influenced by the quality of the soil. Before deciding on the cultivation of a certain variety, one ought to make sure that it is appropriate for the soil at disposal for the purpose. As as rule, light, well-drained soil in an elevated position is to be recommended. West Indian lemongrass yielded 0.31 per cent. of oil on light, sandy, elevated soil and only 0.27 per cent. on low-lying land. In another case, the respective yields were 0.35 and 0.28 per cent.

The second cut is generally richer in oil than the first; in the first year, however, the second cut yields far less grass and therefore less oil than the first.

The parts of the lemongrass which are just above the ground are richest in citral. Large plants were cut into three parts: the top part contained oil with 70 per cent., the middle part, 78 per cent., and the bottom part, 82 per cent. of citral. The moisture of the soil likewise influences the citral contents. Thus, *e. g.*, the same Florida variety of lemongrass yielded oil with 75 per cent. of citral on dry sandy soil, with 68 per cent. on slightly damp sandy clay and with 66 per cent. on moist clayey soil in the neighbourhood of a lake.

Hood is of opinion that lemongrass oil ought to be judged by its citral content and not according to its solubility.

We cannot agree with him in taking no notice of the solubility, but demand now as ever that, in addition to a certain proportion of citral, a good lemongrass oil must show a sufficient solubility in alcohol. Bad solubility indicates the presence of resinified constituents or of unsoluble hydrocarbons, which make the oil useless for certain purposes. As a matter of fact, all attempts of introducing the sparingly soluble oils in commerce have failed, so far. According to the latest investigations, it seems to be pretty sure that the sparingly soluble, so-called West-Indian, lemongrass oil from *Cymbopogon citratus* contains myrcene, which is known to be sparingly soluble and which, above all, has a strong tendency to form insoluble polymerization products. The results of Hood's experiments indicate that exhaustive distillation of lemongrass leads to oils with high citral content and which are readily soluble in the beginning.

The oils obtained in Florida were all well soluble at first, but after three months' storage they had lost their ready solubility.

Unfortunately, Hood does not give any further details as to the physical qualities of the Florida oils.

The costs per acre are as follows: —

first year:		following years:	
preparing the soil	3 dollars		
planting	2 „		
manure	8 „	manure	8 dollars
labour	2 „	labour	1 „
cutting and distillation	5 „	cutting and distillation	8 „
	together 20 dollars		together 17 dollars

takings per acre: { first year = 25 lbs. of oil at 80 cents = 20 dollars; following years = 35 lbs. of oil at 80 cents = 28 dollars.

Several small items not having been taken into consideration, it is questionable whether lemongrass growing by itself will pay. Hood advises, in consequence, to carry it on in connection with other aromatical plants.

On page 29 of our April *Report* **1915** we discussed the occurrence of a white and of a red-stemmed variety of lemongrass, observed in Madras. The distillates from both varieties were duly described by the London Imperial Institute. The oil from the white-stalked grass reminded as well by its smell as by its other qualities more of citronella oil than of lemongrass oil. Recently its botanical examination cleared up[1]) the question of the origin of the mother-plant, which we identified as *Cymbopogon flexuosus*, Stapf *f. albescens*, a colour variety of the common lemongrass. The oil obtained on this occasion showed the following characteristics: d_{150} 0.915; insoluble in 5 vols. of 70 per cent. alcohol at 15°; soluble in 2.5 vols. of 70 per cent. alcohol at 20°; soluble in 0.7 and more vols. of 80 per cent. alcohol. The aldehyde content amounted to 81.0 per cent.

Hence it appears that the oil and its aldehyde content is normal, whereas the oil formerly described only contained 9 per cent. of aldehydes and gave the following data: d_{150} 0.909; α_D — 10° 50′; soluble in 0.8 vols. and more of 80 per cent. alcohol; insoluble in 70 per cent. alcohol. How the bad solubility of the oil could be explained

[1]) *Bull. Imp. Inst.* **14** (1916), 381.

has hitherto remained a mystery to the chemists of the Imperial Institute. Probably the question depends on the manner the product is distilled.

Doubtless also the Formosan lemongrass oil ("Hyang Bow oil") belongs to the category of those so-called West-Indian lemongrass oils (from *Cymbopogon citratus*), which are only soluble with difficulty. According to K. Kafuku[1]), it has the following qualities: $d_{22°}$ 0.8829; α_D + 0.1° to — 0.1°; $n_{D22.5°}$ 1.4850; acid v. 3.1. It contains 66.8 per cent. aldehydes, of which 64 per cent. are citral, and is insoluble in 70, 80 and 90 per cent. alcohol, soluble, however, in 1 vol. of absolute alcohol. The terpene fraction boils from 39 to 39.5° (?) and consists of myrcene.

Nothing is mentioned in the short abstract we have at our disposal about the mother-plant of Formosan lemongrass oil; its qualities, however, lead to the conclusion that it originates from *Cymbopogon citratus*.

The presence of myrcene in its composition fully agrees with former observations made with so-called West-Indian lemongrass oil, according to which the first 20 per cent. of this oil have a specific gravity of 0.821 (in distinction to 0.882 in the East-Indian variety), which indicates the presence of an olefinic terpene[2]). How Kafuku detected the myrcene is not mentioned in the abstract.

Oil of Liquidambar formosana. The genus *Liquidambar*, belonging to the N. O. *Hamamelidaceæ*, numbers four species, of which *L. orientale*, Mill. produces the *Styrax liquidus* of commerce, whereas *L. styraciflуum* is known as the mother-plant of the American storax. A third species, *L. formosana*, Hance, is not only common on Formosa, but also disseminated throughout the whole of Southern China. K. Kafuku[3]) relates in reference to it that from the leaves and twigs of the plant he obtained a yield of about 0.05 per cent. It has a light yellowish-green colour and smells of terpene. Constants: $d_{20°}$ 0.8655; α_D — 3.3°; $n_{D20°}$ 1.4755; viscosity at 20° 1.69; acid value 0; ester value 5.9; ester value after acetylation 25.2. The oil consists principally of terpenes, of which camphene, α-pinene and dipentene were positively identified[4]), whereas the presence of β-pinene and of phellandrene was highly probable. Traces of aldehydes and ketones were also evident which could only be recovered partly from the bisulphite compounds.

This oil differs entirely from the ordinary storax oil produced from the storax-resin of *Liquidambar orientale*, Mill. However, in its physical qualities it shows some resemblance to the product distilled from the leaves of the American storax tree (*Liquidambar styraciflуum*, L.) which has the specific gravity of 0.872 at 15°[5]).

Massoi Bark Oil[6]). From New-Guinea Massoi bark we obtained in a test distillation 8.2 per cent. of a yellow, aromatic smelling oil; $d_{15°}$ 1.0169; α_D + 1° 45′; $n_{D20°}$ 1.52535; soluble in 1.2 vols. of 70 per cent. alcohol; 85 per cent. consisted of eugenol (melting point of the benzoyl compound 69°). The non-phenolic compounds had a specific gravity of $d_{15°}$ 0.9343 and reminded by their odour of nutmegs and of cardamoms. Safrole which had been detected in the oil of massoi bark from New-Guinea on a previous occasion[7]) did not appear to be present in this case, the quantity of oil available, however, was so small that it was impossible to say for certain in this case.

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[1]) *J. Chem. Ind. Tokyo* **19** (1916), 403; *Journ. Soc. chem. Industry* **35** (1916), 865. — [2]) Gildemeister and Hoffmann, *The Volatile Oils*, 2nd ed., vol. II, p. 206. — [3]) *J. Chem. Ind. Tokyo* **19** (1916), 516; *Journ. Soc. chem. Industry* **35** (1916), 906. — [4]) In what manner is not apparent from the abstract. — [5]) See Gildemeister and Hoffmann, *The Volatile Oils*, 2nd ed., vol. II, p. 542. — [6]) Concerning Massoi bark oil vide Gildemeister and Hoffmann, *The Volatile Oils*, 2nd ed., vol. II, p. 489. — *Report* October **1916**, 36. — [7]) *Bericht* October **1888**, 42.

Neroli Oil. On page 39 of our October *Report* **1916** we discussed a paper by R. Guyot[1]) on the green colouring of orange flower water. Guyot[2]) has since continued his investigations and now reports on the morphology and the nutrient mediums of the chromogenous microbes of orange flower water. The temperature most suitable for the development of the microbe is about 20°, between 55 and 60° it is destroyed. The microbe does not form any green crystals on artificial nutrient mediums in distinction to Laseur's *bacillus chlororaphis.* The author explains the fact that the new microbe does not produce the green colouring to any extent on artificial nutrient mediums by the hypothesis that orange flower water is not sufficiently nourishing for it and that in consequence it is compelled to form the green dye, in order to protect itself from violet and ultraviolet rays, which act is a strongly oxidizing manner on it.

Palmarosa Oil. The periodical *Indische Mercuur* copies an article[3]) from the *Indian Trade Journal* on the "Rosha (Rusa) oil industry in British India", in which we find some interesting indications as to the production of palmarosa and gingergrass oils. It is to be regretted that the article almost only mentions "rosha oil", which seems to mean palmarosa oil as well as gingergrass oil[4]), two entirely different products; however, in most cases palmarosa oil seems to be referred to. The treatise is by R. S. Pearson and has been edited by the Forest Research Institute of Dehra Dun.

Rosha oil (from *Cymbopogon Martini*) is also called Nimar oil in India and has, in addition, numerous local names, such as: *"rusha", "rosha", "rohish", "nanha", "duhdi", "sail tall", "mircha", "gandha", "gandi", "tikari", "karva",* &c.

Rosha oil, *i. e.* the "motia" (palmarosa oil) as well as the "sofia" (gingergrass oil), is employed throughout India to make the so-called "attars"[5]). Calcutta, Benares, and other towns in northern India consume for this purpose more than 3000 to 5000 lbs. yearly, the oil being first sent to Bombay and from there farther north. Besides, it is forwarded direct to Burhampur and the Nimar districts, where it is likewise worked up into "attars". Most likely, small quantities are kept in the producing districts and used there, but the relative exact details are wanting. Rosha oil, especially "motia" (palmarosa oil) passes for a remedy against rheumatism and is sold for this purpose in most of the bazaars. Besides, it is used on a small scale for perfuming soaps. The oil is said to possess cooling and astringent qualities and is therefore taken against headache and skin diseases; furthermore, it is supposed to be a remedy against baldness. It is also taken internally against bilious complaints, but in very small doses only.

If the oil is meant to be used as an adulterant of otto of roses, it must first be shaken with a gum arabic solution and then exposed to the sun. Thus the oil gets lighter in colour and agrees in this respect better with the rose oil[6]). Rosha oil which has been heated too strongly in distilling, is said to be no more good for the purpose.

The rosha oil sold in India is rarely pure, but adulterated with oils of turpentine, linseed, colza, or ground-nut, the faking being done in most cases by the distillers.

The quantity of rosha oil exported from India between July 1st, 1912 and June 30th, 1913 amounted to 136263 lbs. As per indications of Volkart Bros., about 4000 lbs. of rosha

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[1]) *Journ. de Pharm. et Chim.* VII. **13** (1916), 37; *Chem. Zentralbl.* **1916**, II. 74. — [2]) *Journ. de Pharm. et Chim.* VII. **15** (1917), 12; *Chem. Zentralbl.* **1917**, I. 225. — [3]) *The Indian Trade Journal; Indische Mercuur* **39** (1916), 787 (N° 35 of September 1st). — [4]) Comp. Gildemeister and Hoffmann, *The Volatile Oils,* 2nd ed., vol. II, p. 178 and 190. — [5]) The native name for perfumes without alcohol and frequently with sandalwood oil as a basis. — [6]) Comp. Gildemeister and Hoffmann, *The Volatile Oils,* 2nd ed., vol. II, p. 556.

oil are consumed in the Nimar district and from 3000 to 4000 lbs. exported from Bombay to north India. Finally, 3000 to 4000 lbs. are consumed in the other producing districts or sent direct to Calcutta, Benares, and the other north Indian towns, so that the total yearly production may come up to 150000 lbs. or so.

Another estimation, originating from the firm of Bauer & Krause, takes the annual production to be 90000 to 100000 lbs., of which 20000 lbs. are consumed in India, 20000 lbs. in Arabia and 60000 lbs. in Europe. This refers, according to Pearson, without doubt to the average production of former years and is too low, therefore. The Bombay Chamber of Commerce does not distinguish between "motia" and "sofia", so that we have at our disposal only the total figures for both.

Exports of rosha oil (palmarosa and gingergrass oils) from British India during the years 1901 do 1913, according to the report of the Bombay Chamber of Commerce: —

year	exports	year	exports
1901/1902	23460 lbs.	1907/1908	57750 lbs.
1902/1903	39580 „	1908/1909	145256 „
1903/1904	161590 „	1909/1910	52148 „
1904/1905	162990 „	1910/1911	59638 „
1905/1906	39950 „	1911/1912	136263 „
1906/1907	44331 „	1912/1913	81660 „
	average . . .	83718 lbs.	

The leading importing countries are: Egypt, France, Germany, the United States, England, the ports on the Red Sea, Turkey and China, whereas Bombay exports small quantities to Calcutta.

Imports of rosha oil (palmarosa and gingergrass oils for the year 1912/1913: —

Great Britain	9750 lbs.,	principally to London,
France	20665 „ ,	„ „ Marseilles, Havre and Dunkirk,
Germany	12800 „ ,	„ „ Hamburg,
United States	6250 „ ,	„ „ New York.
Port Said	14100 „	
Suez	6000 „	
Alexandria	10100 „	
Hongkong	1500 „	
Austria	495 „	

Formerly, the chief producing districts were West Kandesh, Pimpalner, Akrani, Nandurbar, Shahada and Taloda Talukas, whence it was sent in "pakkals" or leather bags to Surat or Bombay *via* Kondabara Ghat, Nawapur and Songat.

On account of experiments made in Chikalda, Pearson arrived at the conclusion that the distillation of rosha oil is not carried out in a rational way. If distilling with steam, grass that has previously been dried in sunshine, 20 per cent. more oil would be obtained than by following the old method of direct heating (?)[1]). If dry fresh grass was distilled in a current of steam, the yield would be doubled, no oil being lost by evaporation as when drying the grass. On an average, and supposing the grass was distilled in a half-withered state, steam distillation would yield about 50 per cent. more oil than the method now in use. In addition, the consumption

[1]) Most likely, distillation with water is meant, for "motia" grass is put into the stills together with water, the heating being done on an open fire. Comp. Gildemeister and Hoffmann, *The Volatile Oils*, 2nd ed., vol. II, p. 181.

of fuel would be considerably smaller with steam distillation than with the old method, and the expenses for wages would be lower too.

The cost of the production of 190 tolas = 4.87 lbs. of oil are 10.13 rupees, if five cart-loads are distilled daily (?). The rent for a modern still is estimated at about 6 annas 3 pies, instead of 3 annas 3 pies for the old installation. The net profits, when using a steam distillation apparatus, are said to be 11 annas daily (?).

The introduction of steam distillation would increase the annual output from about 150000 to 225000 lbs., and the author doubts whether the market can take up this quantity without a considerable fall in prices. A sudden increase of the production, however, seems improbable.

At present, the output is about 150000 lbs. to the value of 750000 rupees, from which the Government receives a royalty of about 45000 rupees.

Patchouli Oil. A patchouli oil of French origin which we received from Switzerland for examination showed the following properties: $d_{15°}$ 1.0171; α_D — 51° 40′; acid value 0.9; ester value 108.3; soluble to any extent in 90 per cent. alcohol. That the sample is evidently adulterated is proved by its far too high specific gravity and ester value, normal products of the kind only showing a specific gravity not exceeding 0.995, and the ester number also is quite low, reaching 1.5 to 12 in oils from Singapore and 6 to 18 in oils from Java. The adulterant proved to be phtalic ester and amounted to 20 per cent. at least. The prices paid at present for pure patchouli oil make such manipulations exceedingly profitable.

Peppermint Oil. It is generally regarded as an accepted fact nowadays that *Mentha piperita* is a hybride between *Mentha aquatica* and *Mentha viridis*. This is also proved by the fact that P. van der Wielen[1]), on sowing commercial seed from *Mentha piperita*, obtained more than 20 different varieties of that plant, but strange to say, not one of them had the characteristic smell of peppermint. Neither did he succeed by means of cuttings, *i. e.*, by vegetative means, in producing peppermint plants in Holland with a pure smell of peppermint; he concluded, however, that the shoots, used for cultivation, did not originate from the right mother-plants.

One frequently finds in literature instructions as to the best manure or fertilizer for peppermint plants. Charabot and Hébert[2]) have particularly studied this question, besides L. Belle[3]) and F. Pilz[4]), and recently the fertilization of peppermint has been the subject of special remarks in an American[5]) technical journal. Shortly after A. Rolet[6]) discussed this question fairly thoroughly. According to his investigations, peppermint plants, owing to their strong leaf-development, demand above all things a lot of nitrogenous manure. In the Maritime Alps, manure from cesspools and sesame cake is principally used, which have a good effect on light, calcareous soils. Pilz[4]) as well as Rolet espacially emphasize the great demand for potash which peppermint plants show.

According to Charabot and Hébert, a harvest of 20000 kg. of green plants deprives the soil per hectare of 50 kg. nitrogen, 30 kg. phosphoric acid, and 160 kg. potash;

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[1]) *Pharm. Weekblad* **53** (1916), 872. — [2]) *Bull. Soc. chim.* III. **27** (1902), 204; *Report* October **1902**, 67. — *Compt. rend.* **136** (1903), 160; *Report* April **1903**, 61. — *Compt. rend.* **136** (1903), 1009, 1678. — *Bull. Soc. chim.* III. **29** (1903), 612, 698; *Report* October **1903**, 57. — [3]) *Journ. Parfum. et Savonn.* **20** (1907), 80; *Report* October **1907**, 74. — [4]) *Zeitschr. f. landw. Versuchswesen in Österreich* **15** (1912), 575; *Report* October **1912**, 91. — [5]) *Oil, Paint and Drug Reporter* 88 (1915), N° 23, 11; *Report* October **1916**, 43. — [6]) *Parfum. moderne* **9** (1916), Suppl. p. 98.

according to Mazzaron, of 64 kg. nitrogen, 40 kg. phosphoric acid, and 137 kg. potash; according to other investigators, of 140 kg. nitrogen, 40 kg. phosphoric acid, and 120 kg. potash. The gives a mean of 84 kg. nitrogen, 37 kg. phosphoric acid, and 139 kg. potash.

Rolet recommands as artificial fertilizers Chili saltpetre, ammonium sulphate, superphosphate, basic slag powder, potassium chloride and potassium sulphate.

Superphosphate and potash are laid out on the fields together with stable manure. Ammonium sulphate is added before planting, and Chili saltpetre partly before planting, partly during the growth of the plants.

Superphosphate is suitable for soils rich in lime, basic slag powder for those deficient in that substance; the question, in what manner phosphoric acid is to be employed, has not been decided yet.

Potassium chloride is only to be recommended for calcareous soils and must be spread out in autumn or winter; potassium sulphate can be employed everywhere.

In the South, each hectare of siliciferous clay soil is fertilized with 20000 kg. of stable manure or 1500 kg. of sesame cake, besides 300 kg. of Chili saltpetre or 225 kg. of ammonium sulphate, 400 kg. of superphosphate, and 300 kg. of potassium sulphate (500 kg. if sesame cake is used instead of stable manure). The Chili saltpetre is added in 2 portions, once during the cultivation of the fields (200 kg.) and again in the middle of May; ammonium sulphate is only employed in planting. Loamy calcareous, pervious soil on sloping banks demands 300 kg. of Chili saltpetre, 1000 kg. of superphosphate and 300 kg. of potash, which corresponds to an expense of 260 fr.; the yield amounts in this case according to Autran to 20500 kg. of peppermint herb, which are estimated at 12 fr. per 100 kg.

Other instructions recommend 30000 kg. of stable manure or 2500 kg. of sesame cake, 300 kg. of Chili saltpetre, 400 kg. of 16 to 18 per cent. superphosphate, 100 kg. of potassium sulphate, all the above ingredients to be added before planting, with the exception of the Chili saltpetre, which latter must be ploughed into the soil in April.

A rich soil with average lime content demands 300 kg. of sesame cake (containing 5 to 6 per cent. nitrogen and 4 per cent. phosphoric acid), and 120 kg. of potassium chloride (costs, 350 fr.).

A soil, free from lime, requires 1250 kg. of powdered dry blood (containing 12 to 13 per cent. nitrogen), 250 kg. of slag meal (15 per cent.) and 130 kg. of potassium sulphate (46 per cent.). The expense amounts to 300 to 350 fr. Otherwise, one can also employ, 18000 kg. of stable manure, or 950 kg. of sesame cake (7 per cent. nitrogen), or 400 kg. of Chili saltpetre; 300 kg. of superphosphate or of basic slag (16 to 18 per cent.), and 250 kg. of potassium sulphate. Another prescription runs as follows: stable manure, 400 kg. of superphosphate, 300 kg. of potash, 300 kg. of gypsum: in April, additional 300 kg. of Chili saltpetre and 300 kg. of gypsum. Or, stable manure, 300 kg. of superphosphate, 100 kg. of potassium chloride and 200 kg. of Chili saltpetre, the latter in the spring.

In testing the peppermint spirit frequently used in North America it is not sufficient to determine its content of peppermint oil, on the contrary, one must also try to ascertain the quality of the oil, because sometimes oils are employed for manufacturing the spirit which have been freed from menthol or even adulterated. Following the directions of the American Pharmacopœia spirit of peppermint should be produced by treating peppermint leaves with alcohol which have been previously soaked in water. According to H. L. Thompson[1]), the test for examining peppermint spirit on

[1]) *Americ. Journ. Pharm.* 88 (1916), 303.

its content of free or esterified menthol is performed in the following manner: 50 ccm. peppermint spirit are filled into a measuring flask holding 50 ccm. and weighed; hereupon the spirit is transferred into a small cassia-flask, rinsing the flask three times with 10 ccm. of water, water added up to the mark, and the vessel allowed to stand at least 4 hours. The quantity of oil which has separated is read off and the oil yield of the spirit calculated, expressed in per cent. by volume.

One or 2 ccm. of the oil are then poured into a previously weighed cylinder which besides is divided into tenths of a ccm. and the content of peppermint oil in the spirit calculated from the weight obtained, expressed in per cent. by weight. The oil remaining in the neck of the small cassia-flask is then carefully removed and the aqueous liquid employed for ascertaining its alcohol content.

The oil remaining in the tared cylinder is transferred into an Erlenmeyer-flask, weighed and saponified, in order to determine is menthyl acetate content.

The saponified oil is now acetylated and saponified to ascertain its menthol content. In order to determine the alcohol content of the spirit of peppermint the watery-alcoholic liquid from the small cassia-flask is poured into a fractionating flask and the cassia-flask rinsed with 100 ccm. of water. Finally, after distilling not quite 100 ccm. of this liquid into a measuring flask of 100 ccm., one fills up to 100 ccm., determines the specific gravity of the liquid, and calculates therefrom its alcohol-content.

Peppermint Oil, American. The influence of climatic conditions or the quality of the soil are frequently adduced as reasons for diversity in the quality or composition of volatile oils. Experiments with this object in view have only rarely been made, and it is very doubtful if the variations which have been observed can always be explained by the causes mentioned above. A publication by F. Rabak[1]), which has been issued by The Bureau of Plant Industry of the U. S. under the designation of "The Influence of cultivation and Climatic conditions on the Yield and Quality of Peppermint oil", is well worth our attention. He cultivated peppermint plants under various climatic conditions on soils differing widely and distilled the material obtained from plants in various stages of development, besides trying to arrive at definite conclusions by not only distilling the entire plants, but also in parts, in a dry as well as in fresh state. The plants were cultivated at Webster, South Dakota, on a dark, fertile, loamy soil, at Glenn Dale, Md., on a light, sandy soil, and at Arlington Farm, Va., on a heavy, clay soil. The results obtained proved that the quality of the soil had very little effect indeed on the yield, the same applied to the colour, smell, and taste of the oils, whereas the optical rotation and refraction showed very great differences. The oil from Arlington (fertile, loamy soil) was specifically lighter and rather more soluble in 80 per cent. alcohol than the others. The Arlington oil contained less esters and a smaller amount of total menthol than that from Webster, whereas the oil from Glenn Dale was the richest in esters.

This and the investigations of other authors[2]) leads Rabak to the conclusion that light, sandy or loamy soils are more favourable for the production of esters and menthol in peppermint oil than soils of a heavy nature. Climate seems to have very little effect on the production of the oil. Table I gives the results concerning qualities and yields in oil from plants in various soils.

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[1]) *U. S. Dep. Agric. Bull.* No 454, Washington 1916. — [2]) H. J. Henderson, *Pharmaceutical Journ.* **87** (1911), 175; *Report* October **1911**, 70. — J. C. Umney, *Pharmaceutical Journ.* **57** (1896), 103; *Report* October **1896**, 59. — E. Charabot and A. Hébert, *Compt. rend.* **136** (1903), 160. — G. Mossler, *Über den Einfluss verschiedener Kulturbedingungen auf das ätherische Öl von Mentha piperita, Pharm. Post* **45** (1912), 2.

In order to study the influence of the time the plants were harvested and the quality of the material which was to be distilled, Rabak treated the plants not only in the fruiting period, but also before and after they blossomed, in a fresh as well as in a dry state. Whereas the oils from budding and from flowering plants showed varying properties, those gathered from fruit-bearing plants were more uniform in quality. The dried herb (with due regard for loss in weight) produced 50 per cent. less oil than fresh, hence drying before distillation, according to Rabak, is to be avoided. On the whole it appeared that the oil yield diminishes the more the plant approaches maturity. The herb in a whole state produced less oil than the leaves and stalk ends; the leaves contain most oil, the stalks hardly any. On the average the oil yield from fresh, budding herb amounted to 0.134 per cent., from dry herb to 0.66 per cent., from fresh flowering herb to 0.132 per cent. from dry ditto to 0.050 per cent., finally from fresh, fruit-bearing plants to 0.114 per cent., and from dry ditto to 0.046 per cent.

From the entire fresh plants he obtained on distilling when budding 0.116 per cent., during the blossom stage 0.113 per cent., and while bearing fruit 0.133 per cent. The leaves of plants produced before blossoming and distilled in a fresh state gave on an average 0.203 per cent. of oil, those from blossoming plants 0.303 per cent., and those from plants bearing fruit 0.120 per cent. The tops, gathered from budding plants, contained in a fresh state on an average 0.173 per cent. of oil, those from flowering plants 0.233 per cent., and those from fruit-bearing plants 0.153 per cent.

Apparently the various vegetation stages of the plant itself have very little effect on the colour, smell, or taste of the oil. Oils from flowering plants are specifically heavier and show a lower rotation than those from plants before blossoming or in a fruit-bearing stage. The oils vary exceedingly in reference to their solubility in 80 per cent. alcohol. Oil from dry plants is generally darker in colour than that from fresh plants and also has a less agreeable smell. The ester-content increases with the development of the plant. Oils rich in esters frequently contain only little free menthol and *vice versâ*. Oils from dry plants are richer in free acids and esters than those from fresh plants; drying seems to have a favourable effect on the production of esters; apparently malodorous compounds, however, are formed thereby which produce the bad smell, referred to above, of oils from dry plants. The formation of

Table I.

Yields and properties of peppermint oils grown on various soils.

Locality and quality of soil	Yield	$d_{22°}$	α_D	n_D	Solubility in 80 per cent. alcohol	acid [1] content	ester [2] content	Menthol content free	Menthol content total
	per cent.					per cent.	per cent.	per cent.	per cent.
Arlington Farm (heavy, clay soil	0.12 and 0.13	0.9203 and 0.9273	−21.2 and −11.4°	1.4662 at 25° and 1.4820 at 22°	in 1.2 vols., becomes turbid in 2 vols., and 0.6 vol., becoming turbid in 2.5 vols.	0.21 and 0.03	7.07 and 11.67	46.5 and 30.94	51.7 and 40.13
Webster (rich black loam)	0.10	0.9280	−16°	1.4715 at 25°	in 1.5 vols., becomes turbid in 2.5 vols. and more	0.05	9.9	46.48	54.28
Glenn Dale (light, sandy soil)	0.11	0.9403	−20°	1.4735 at 23°	in 0.8 vol., becomes turbid in 2.5 vols. and more	0.75	23.34	35.89	54.28

[1]) Calculated as free acetic acid. — [2]) Calculated as menthyl acetate.

Table II.

Physical and chemical properties of oils taken from fresh and dry herb in its various stages of development.

Stage of development	d	α_D	n_D	acid[1]) content per cent.	ester[2]) content per cent.	Menthol free per cent.	Menthol total per cent.
			A. Oil from fresh plants.				
budding	0.912 at 23° to 0.9158 at 22°	— 18.4 to — 31.6°	1.4696 at 24° to 1.4738 at 25°	0 to 0.86	6.72 to 16.62	30.1 to 43.76	35.39 56.85
flowering	0.9203 at 22° to 0.9273 at 24°	— 9 to — 21.2°	1.4662 at 25° to 1.4820 at 22°	0 to 0.21	7.07 to 14.5	23.97 to 46.5	35.39 52.09
fruiting	0.9151 at 22° to 0.9250 at 24°	— 14 to — 42°	1.4676 at 25° to 1.4712 at 22°	0 to 1.14	12.37 to 24.4	12.7 to 34.33	29.14 53.55
			B. Oil from dry plants.				
budding	0.9167 at 24° and 0.9337 at 21°	— 22 and — 26.2°	1.4695 at 25° and 1.4724 at 22°	0.17 and 1.20	13.26 and 19.80	36.07 and 39.41	46.38 and 55.01
flowering	0.9163 at 22°	— 27°	1.4695 at 25° and 1.4765 at 22°	0.16 and 3.20	12.73 and 30.41	37.37	47.44
fruiting	0.9344 at 22° and 0.9383 at 24°	— 6.6 and — 16.2°	1.4713 at 22° and 1.4718 at 25°	0.36 and 0.56	17.68 and 20.51	34.2	48.15

Table III.

Physical and chemical properties of the oils obtained from various parts of fresh plants.

Oil from	d	α_D	n_D	acid[1]) content per cent.	ester[2]) content per cent.	Menthol free per cent.	Menthol total per cent.
			A. Plant budding.				
Whole plant	0.9155 at 24° and 0.9158 at 22°	— 18.4 and — 24.2°	1.4718 at 22° and 1.4738 at 25°	0.1 and 0.86	6.72 and 16.62	30.1 and 43.76	35.39 and 56.85
Leaves	0.9143 at 22° and 0.9156 at 24°	— 24.2 and — 26.2°	1.4710 at 22° and 1.4713 at 25°	0.014 and 0.9	9.9 and 19.80	18.79 and 37.7	34.39 and 45.67
Tops	0.9151 at 22°	— 24°	1.4718 at 25°	0.116 and 0.13	8.84 and 13.44	29.19 and 31.8	38.76 and 39.78
			B. Plant flowering.				
Whole plant	0.9203 at 22° and 0.9273 at 24°	— 11.4 and — 21.2°	1.4662 at 25° and 1.4820 at 22°	0.03 and 0.21	7.07 and 11.67	30.94 and 46.5	40.13 and 52.09
Leaves	0.9231 at 24° and 0.9389 at 22°	— 18.8 and — 22°	1.4706 at 25° and 1.4753 at 22°	0.52 and 1.90	17.3 and 22.27	37.83 and 41.7	55.38 and 55.38
Tops	0.9242 at 24° and 0.9370 at 22°	— 13 and — 20.6°	1.4732 at 25° and 1.4780 at 22°	0.48 and 1.43	15.9 and 22.98	37.75 and 48.60	50.29 and 66.71
			C. Plant fruiting.				
Whole plant	0.9151 at 22° and 0.9250 at 24°	— 14 and — 16.6°	1.4676 at 25° and 1.4712 at 22°	0.10 and 1.14	12.37 and 20.86	12.7 and 33.8	29.14 and 43.58
Leaves	0.9373 at 22°	— 19.2°	1.4730 at 25° and 1.4720 at 22°	0.32 and 4.8	19.8 and 43.40	37.58	53.18
Tops	0.9223 at 22°	— 19°	1.4723 at 25° and 1.4738 at 22°	0.16 and 3.30	15.56 and 28.29	34.8	47.08

menthol and of menthyl acetate seems to be restricted principally to the stalk ends. Table II gives information concerning the qualities of the oils from dry and fresh plants during the various stages of vegetation, whereas table III refers to the qualities of the oils produced from the different parts of the plant.

[1]) Calculated as free acetic acid. — [2]) Calculated as menthyl acetate.

Rabak further tried to form an estimate in what manner shading had any effect on the production of peppermint oil and on its qualities. For this purpose he not only planted peppermint on a bed on which the intensity of light was reduced about 50 per cent. but also on one of the same size which remained unshaded[1]).

The oil yield was about the same; on the whole the oils from both beds showed little difference. Oil from plants grown in the shade contained rather less acid, menthyl acetate, free menthol, and total menthol than oil from plants reared in full daylight. Accordingly it appears that the formation of esters as well as of menthol is retarded by shading. The properties of the oils were the following: —

Oil from herb grown in full daylight: $d_{23°}$ 0.9233 and 0.9273; α_D — 11.4° and — 14.6°; $n_{D23°}$ 1.4722 and $n_{D22°}$ 1.4820; acid content (calculated as acetic acid) 0.03 and 0.20 per cent.; ester content (calculated as menthyl acetate) 11.67 and 13.79 per cent.; free menthol 30.94 and 34.11 per cent.; total menthol 40.13 and 44.97 per cent.

Oil produced from shade-grown plants: $d_{23°}$ 0.9192 and 0.9343; α_D — 4.48 and — 6.8°; $n_{D23°}$ 1.4708 and $n_{D22°}$ 1.4724; acid content (calculated as above) 0 and 0.07 per cent.; menthyl acetate 10.96 and 13.08 per cent.; free menthol 24.43 and 31.20 per cent.; total menthol 33.07 and 41.50 per cent.

Finally, Rabak also mentions the effect of frost on the quality of the peppermint oil. Oil from frozen plants smells pleasanter and more flowery, probably owing to the high menthol and ester content; hence it is also comparatively easily soluble and may perhaps contain less terpenes[2]).

The oil produced from frozen plants showed the following properties: — $d_{24°}$ 0.9107 and $d_{23°}$ 0.9155; α_D — 33 and — 23°; $n_{D23°}$ 1.4580 and $n_{D25°}$ 1.4592; acid content (calculated as above) 0.09 per cent.; menthyl acetate 40.66 and 24.3 per cent.; free menthol 28.93 and 40.7 per cent.; total menthol 60.97 and 67.8 per cent.

Rabak's statements concerning the oil yields from dry and fresh plants are partly at variance with the observations of other scientists and with the results of practical experience[3]). We will not omit to mention, however, that comparative experiments on the decline of the yield in volatile oil during the process of drying plants are exceedingly difficult, especially in the case of such containing much water. Losses up to 50 per cent. of oil during drying, as Rabak mentions, may well be caused by inappropriate treatment of the herb during the drying process. Before the fact was known that fresh material for distillation easily retains part of the oil, scientists even imagined that the oil content increased during drying, till exhaustive distillation tests proved that this idea was altogether erroneous. Fresh distillation material produces, when completely distilled, a yield in oil differing little or not at all from that produced by the same initial substance when distilled in a highly faded state, calculated on fresh plants, on the condition that the drying process is performed in an appropriate manner. The realisation of the fact that fresh plants can only be distilled completely with much loss of time and steam, owing to repeated interruption of the distillation process, in order to enable the contents of the still to get dry, has lead to the result that in practice[4]) peppermint plants are almost invariably distilled in a highly faded or dry state, whereby the highest yield in oil is obtained with the least expense.

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[1]) H. J. Henderson obtained, on the contrary, from plants on sunny sites 0.409 per cent. of oil and from plants on damp, shady spots only 0.1 per cent. of oil. (Gildemeister und Hoffmann, *Die ätherischen Öle*, 2nd ed., vol. III, p. 578.) — [2]) A similar effect has also been observed in the case of bergamot mint oil (*Mentha citrata*, Ehrh.), the product obtained from frozen herb containing much more linalyl acetate than otherwise. (Gildemeister und Hoffmann, *Die ätherischen Öle*, 2nd ed., vol. III, p. 590.) — [3]) v. Rechenberg, *Theorie der Gewinnung und Trennung der ätherischen Öle durch Destillation*, p. 278 and 279. — [4]) Comp. preceding note, p. 461.

Besides the oils, when distilled from fresh or dry plants, differ accordingly to a considerable extent[1]). When fresh plants are used for distillation, the process takes some time and resinous compounds easily get into the oil, which otherwise when dry material is employed become oxidized by the oxygen of the air into non-volatile resins. Accordingly, oils produced from dry herb do not become resinous so easily and are more stable[2]), whereas the oils distilled from fresh plants have first to undergo a process of resinification, whereby their solubility easily suffers and their specific gravity increases.

Peppermint Oil, Japanese. In an article by A. Dyes[3]) on Greater Japan's economical development, taking into consideration the chemical and allied industries, we find some figures concerning the peppermint production. Judging by them, the export of menthol crystals would be increasing considerably. The figures for 1913 and 1914 ran as follows: —

Menthol crystals	1913	1914
British India	151	106
Great Britain	767	276
France	357	119
Germany	1015	582
United States	470	625
other countries	101	105
	total: 2872	1816[4])

The production amounted to: —

	crude peppermint (?)[5])		peppermint oil		menthol	
	lbs.	dollars	lbs.	dollars	lbs.	dollars
1908	141 090	114 570	89 364	93 182	79 268	107 978
1909	228 711	248 208	143 317	156 255	149 103	263 096
1910	248 745	382 430	158 243	177 261	150 413	276 875
1911	352 960	728 807	165 356	222 887	166 161	295 933
1912	619 512	1 553 303	208 128	296 537	202 457	999 278

In Japan, the yield of dried peppermint leaves amounts to 243 to 375 lbs. per acre and varies in the different districts. On an average, the annual production from 1908 to 1912 was 321 lbs. per acre. In above American statistics, the "crude peppermint"(?)[5]) does not include peppermint oil and menthol. One reckons about 12 cents of costs for 100 lbs. of plants and 495 lbs. of plants per acre; hence, the costs per acre would be 60 cents or so. The area planted with peppermint came up to 11 373 hectares, in 1913, and the leaves obtained weighed 42 000 tons or 11 million kwans. The increase compared with former years is most surprising. The area planted was: —

	in hectares (cho)	in kins		in hectares (cho)	in kins
1910	3010	3 031 737	1912	6 355	6 344 030
1911	4177	4 052 723	1913	11 373	11 076 006

In 1914, the Hokkaido planters transferred their rights of selling to Samuel, Samuel & Co., which called forth the opposition of the Japanese forestallers.

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[1]) Gildemeister und Hoffmann, *Die ätherischen Öle*, 2nd ed., vol. III, p. 550, 551. — [2]) v. Rechenberg, *loc. cit.*, p. 281. — [3]) *Chem. Ztg.* **41** (1917), 518. — [4]) To be understood in thousands of yen, as per a communication by letter from the author. — [5]) It is not at all clear to us what the author means by "crude peppermint".

End of March 1917, the price of menthol was 12 s. 6 d. per lb. in London. The menthol exports from Japan were as follows: —

	1914	1915	1916
kins . . .	269266	325464	386458
yen . . .	1816096	1805114	2410628

The countries of destination were: —

	1914	1915	1916	
British India	13906	26177	24707	kins
Great Britain	42720	59246	143148	„
France	22329	41774	69184	„
Germany	82600	—	—	„
United States	89496	172590	137228	„
other countries	18215	25677	12191	„
total:	269266	325464	386458	kins.

According to an English trade journal[1]), the 1916/17 crop of menthol and peppermint oil taken together is estimated at 1659998 lbs., against 1406655 lbs. in the preceding year. Owing to the drought, one reckons in Hokkaido to be 30 or 40 per cent. short of the output estimated at more than 1000000 lbs. The 1915/16 crops were, and the estimations for 1916/17 are, the following for the various districts: —

	1915/16	1916/17
old stocks in the producing districts	200000 lbs.	106666 lbs.
production in the Sambi district	133333 „	273333 „
„ „ „ Yamagata „	6666 „	6666 „
„ „ „ Hokkaido „	866666 „	1000000 „
old stocks in Kobe and Yokohama at the beginning of July	200000 „	273333 „
total:	1406665 lbs.	1659998 lbs.

Peppermint Oil, Saxon. As mentioned already several years ago[2]), our climate is hardly suitable for growing peppermint, a plant so sensitive towards cold and pests, so that our troublesome and expensive experiments in this line afforded us but very little satisfaction. The last severe winter had such a destructive effect on the plants that the few remaining peppermint fields were completely done for. We have, in consequence, definitely given up this branch of our Miltitz cultivation. We would refer all those interested in this kind of peppermint oil to our quality "Miltitz 1914"[3]), introduced in 1914 and which can without hesitation be recommended as a perfect substitute for the Saxon oil.

Pine Needle Oils. The oil from pine, red fir, or "Norway spruce" needles also belongs to the volatile oils occurring rather rarely. It is produced from the needles and young twig ends of *Picea excelsa,* Lk. (*P. vulgaris,* Lk.). Hence a few samples may be mentioned here which we received in the course of the year. A distillate from the Black Forest gave the following results: d_{15° 0.8754; α_D — 39° 50′; acid v. 0.6; ester v. 20.5 = 7.2 per cent. bornylacetate, soluble in 5.5 vols. and more of 90 per cent. alcohol.

Two Thuringian oils showed the following constants: d_{15° 0.8795 and 0.8807; α_D — 30° 15′ and — 26° 45′; acid v. 1.9; ester v. 18.7 = 6.5 per cent. bornylacetate, and 16.8 = 5.9 per cent. bornylacetate; soluble in 5 — 6 vols. and more of 90 per cent. alcohol.

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[1]) *Chemist and Druggist* 88 (1916), 1055. — [2]) *Report* October **1913**, 88. — [3]) *Report* October **1915**, 31.

The smell was in each case pleasant and strong and reminded of that of pine needle oil.

According to an official report of the United States Forest Service[1]), 40 to 50000 pounds of "White" and "Black" Spruce fir oil and Hemlock fir oil are produced yearly in the U. S.[2]).

The production of "Red cedar oil" (from *Thuja plicata*, Lamb.?) amounts to 15 to 20000 pounds. This oil and that from "Silver fir" are much used for perfuming soaps as well as for the production of liniments and of other medicinal preparations. Cedar oil is also employed for liniments and for the production of insecticides.

Fatty oil from fir seeds. Fir seeds range among those distinguished by containing fatty oil, which may perhaps be used for the manufacture of edible oil. As per a communication of Count Leiningen[3]), pine and larch seeds would come too expensive to be used for producing oil; the seeds of the silver fir contain drops of turpentine in the covering tissue, so that the oil would get a resinous taste; in consequence, the spruce fir alone of all our native conifers might supply an edible oil.

The cones ought to be gathered from the trees before opening, if possible, and sent to a *Klenganstalt*, where they are dried artificially, when the seeds fall out. As the wings of the seeds consist of dry tissue, full of air-cells, which would absorb oil during the pressing process, it is necessary to remove them, which can be done without trouble, as they come off easily. Already in times of peace, coniferous seeds have been used in large quantities for obtaining oil, which, however, seems to have been of inferior quality.

As the oils from conifers belong to the drying oils, they cannot be used as lubricators, hair oil and all such purposes as demand oils that show but little tendency to dry.

Some years ago, when distilling seeds by themselves, we have shown already that coniferous seeds are sometimes rich in volatile oils[4]). Whilst the entire seeds only yielded 2.3 per cent. of oil, the crushed ones gave off 12 to 13 per cent.

Rose Oil. It is not surprising nowadays that rose oil adulteration should be so much in vogue. Of 3 samples we received recently for examination not even one was up to standard. The constants of the 3 oils are evident from the following table, to which we have added the limiting values of a good Bulgarian trade sample for the sake of comparison: —

		1	2	3	Limits:
Original Oil.	$d_{30°}$	0.8518	0.8538	0.8546	0.849 to 0.862
	α_D	— 0° 44′	— 0° 40′	— 1° 30′	— 1 to — 4°
	$n_{D25°}$	1.45222	1.44962	1.45162	1.452 to 1.464
	Solidification point	+ 23°	+ 21°	+ 21.5°	+ 18 to + 23.5°
	Acid value	1.9	1.9	0.9	0.5 to 3
	Ester value	45.7	31.7	7.5	7 to 16
After shaking with water.	$d_{30°}$	0.8528	0.8563	0.8584	66 to 75 per cent.
	$n_{D25°}$	1.45482	1.45532	1.45992	total geraniol.
	Ester value after acet.	212.8	235.2	231.5	
	Total geraniol content	69.6 per cent.	78.5 per cent.	77 per cent.	

[1]) *Oil, Paint and Drug Reporter; Perfum. Record* **7** (1916), 258. — [2]) The young sprouts of *Abies canadensis* (Hemlock Fir), *Picea alba*, Lk. ("White Spruce" Fir) and *Picea nigra*, Lk. ("Black Spruce" Fir) are often distilled in North America, where the different species are frequently mixed. — [3]) *Zeitschr. f. Abfallverwertung* **1917**, N° 1; *Seifensieder-Ztg.* **44** (1917), 77. — [4]) *Report* October **1912**, 94.

The samples 1 and 2 gave such a high ester value that there is absolute proof of adulteration, in point of fact by the ester of a difficultly volatile acid, especially as the difference between the saponification value and the acid value II[1]) is much too high, whereby it is quite irrelevant whether the ester was added for itself or as a component of an oil. Closer examination revealed nothing particular on this point; anyhow it grew evident that this was not the only adulterant in each case. The increase of the specific gravity after shaking with water proved that the oils contained alcohol, which we were able to eliminate and to identify by converting it into acetic ester. The quantity which had been added amounted to a little over 3 per cent. in sample 1, and 7 per cent. in sample 2. Furthermore, the total geraniol content in the latter is too high, which leads to the conclusion that it had also been adulterated with palmarosa oil, geraniol, or similar substances.

Apparently in sample 3 alcohol was the only adulterant, but to such an extent that the addition amounted at least to 10 per cent. Possibly palmarosa oil was also present, as the oil, after freeing from alcohol, showed a total geraniol content of 77 per cent., giving sufficient ground for suspicion.

Finally, an oil may be mentioned which was offered to us for sale from Constantinople. The sample was so small that only the rotation and solidification point could be determined, which was, however, quite sufficient to give an idea of its quality. The rotation amounted to $-28°\,30'$, and the solidification point to $+27°$! The smell also reminded much more of geranium oil than of rose oil; hence it appeared mainly to be a highly adulterated geranium oil which the seller in his innocence had taken for rose oil, as we do not imagine that he wanted to defraud us intentionally.

The Rose Oil Industry in Asiatic Turkey (Anatolia).

By Dr. G. Bredemann,
Agricultural Expert to the Imperial Government of German New Guinea, now at Constantinople.

Rose growing in Turkey is of fairly recent date. It was introduced in 1894 by a Turkish rose farmer who had immigrated from Bulgaria and smuggled rose plants from Kazanlyk across the Turkish frontier. He started with them on his own account and on a small scale, but soon taught and extended rose growing in the country by order of the Government. He is still now in the Government's service as a specialist for rose growing, which he succeeded, during 20 years of activity, in developing from the most modest beginnings to its present rather considerable extension.

Statistics for the year 1329 (1913) as to the extent of rose growing in European and Asiatic Turkey were published recently by the Turkish Ministry of Agriculture. It is to be found in the "Agricultural Statistics of the year 1329" (Constantinople, Library Osmanié, 1332), edited by the General Governing Board for Statistics of the Ministry of Commerce and Agriculture. This official summary is reproduced on the following two pages 44 and 45:

Attention must, however, be called to the fact that some rather considerable mistakes have crept into this review. Especially the figures given for the area under cultivation in the Vilayet of Aïdin and also in the Vilayet of Konieh are far too high, so

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[1]) Concerning acid values, see Gildemeister und Hoffmann, *Die ätherischen Öle*, 2nd ed., vol. III, p. 75 to 77; *Report* October **1910**, 62.

that the total of 11979 dönums (1 dönum = 919 square metres) is to be reduced in consequence. According to a very cautious estimation, at present there are about 4500 dönums planted with roses, in the whole of the European and Asiatic parts of Turkey. They are distributed as follows: —

Table I.

Review of the area now planted with roses in Turkey.

Vilayet or Sandjak (respectively) of:		dönums:
1. Konieh		2980 to 3000
of which: Sandjak of Konieh		50 to 60
sandjak of Isparta	Kasa Isparta	800
	„ Uluburlu	300
	„ Karaghatch	500
	„ Jaiovatch	150
sandjak of Burdur	„ Burdur	1100
	„ Tefenni	80 to 90
2. Brussa		520
3. Aïdin (vilayet)		400
of which: sandjak of Denisli	Kasa Bulladan	28
	„ West of Karagatch	74
	„ Denislü	13
	„ Tawass	5
	„ Djal	12
4. Kutahia		40 to 45
of which: Kasa Kutahia		30 to 35
„ Ushak		10
5. Afion-Karahissar		236
of which: Kasa Afion-Karahissar		5
„ Diner		225
„ Tchivril		5
„ Sandykly		1.5
6. Dardanelles with Karabigha		40
7. Angora		6
8. Kastamui		9
9. Boli		25 to 30
10. Karassul		10
11. Tekké (Adalia)		15
12. Mentcher (Mughla)		40
13. Diarbekir		100 to 120
14. Marash		20
15. Mosul		25 to 40
16. Syria		50
17. Adrianople		40
		total: 4416 to 4581

These figures do not claim to be absolutely exact, but they have been most carefully estimated. I am indebted for them to a Turkish inspector of rose growing, who in the previous year had personally surveyed the rose fields in the main district, and to the agricultural directors in Kutahia, Afion-Karahissar, Brussa, and Denislü.

It is said that in each of the two last years about 1000 kilos of rose oil, in a round figure, were obtained, a result which supposes a good flower crop and a good yield in oil, taking into consideration the area of 4500 dönums, as indicated.

Review of the area planted with roses in Turkey in the year 1329 (1913) and its yield.

Table II. (as per the "Agricultural Statistics for the year 1329", published by the General Governing Board for Statistics of the Ministry of Commerce and Agriculture. Constantinople, Library Osmanié, 1332 [1916]).

Name of the district		Area planted	Rose leaves			Rose oil			Rose water		
			yield	average price		yield		average price	yield	average price	
		Dönums[1]	Okas[2]	Piasters	Centimes[3]	Okas	Dirhöms[4]	Piasters	Okas	Piasters	Centimes
Vilayet of Aïdin.											
Sandjak of Denislü:	Kasa Bulladan	5000	—	—	—	—	—	—	5000	2	—
„ „ Smyrna:	„ to the West of Karaghatch	20	4000	1	—	—	300	20000	400	6	—
	„ Tire	12	2500	1	—	—	250	20000	500	3	—
„ „ Aïdin:	„ Aïdin	120	40000	1	50	3	—	8000	21000	3	—
	„ Bosdogan	100	—	—	—	3	—	7500	3000	1	50
„ „ Manissa:	„ Kassaba-Torgutlu	60	—	—	—	—	—	—	48000	2	—
	total:	5312	46500	1	16	7	150	15916	77900	3	—
Vilayet of Brussa.											
Sandjak of Brussa:	Kasa Brussa	117	43750	2	—	13	267	16000	21000	—	75
	„ Ohau-Gasi	5	75	2	50	—	150	12000	5	2	—
	„ Kirmasti	—	—	—	—	—	—	—	150	8	—
„ „ Erthogröl:	„ Bjledjik	60	3000	2	—	1	50	5068	500	1	50
	„ Inegöl	100	25000	2	—	7	324	16000	19000	—	75
	„ Sögüd	50	5000	2	—	1	350	16668	2000	2	50
	total:	325	76825	2	10	24	341	13289	42655	2	58
Vilayet of Konieh.											
	Kasa Konieh	50	—	—	—	—	300	8000	—	—	—
	„ Eregli	25	—	—	—	—	—	—	500	2	—
	„ Ermenek	20	—	—	—	—	—	—	—	—	—
	„ Akshehir	5	—	—	—	—	—	—	—	—	—
Sandjak of Isparta:	„ Isparta	1350	—	—	—	1	—	21000	3600	2	—
	„ Uluburlu	900	—	—	—	7	200	21000	1000	2	—
	„ Karaghatch	20	—	—	—	1	100	21000	4000	3	—
	„ Jalovatch	188	—	—	—	4	—	21000	2500	2	—
„ „ Burdur:	„ Burdur	1600	—	—	—	150	—	13868	800	2	—
	„ Tefenni	115	—	—	—	4	—	16000	2000	2	—
	total:	4273	—	—	—	168	200	14648	14400	2	—

Independent Sandjak of Tekké.										
Kasa Adalia	20	—	—	—	—	150	10668	550	4	—
„ Elmaly	17	1300	2	—	—	130	11200	1500	1	50
„ Kakudeli	156	—	—	—	1	169	12000	5000	1	50
„ Alaja	20	—	—	—	—	—	—	100	3	—
total:	213	1300	2	—	2	49	11289	7150	2	50
Independent Sandjak of Afion-Karahissar.										
Kasa Afion-Karahissar	50	—	—	—	—	—	—	—	—	—
„ Diner	1500	227500	3	—	24	150	13000	19500	2	50
total:	1550	227500	3	—	24	150	13000	19500	2	50
Independent Sandjak of Kutahia.										
Kasa Kutahia	18	—	—	—	—	—	—	—	—	—
„ Ushak	35	—	—	—	—	—	—	10000	2	50
total:	53	—	—	—	—	—	—	10000	2	50
Vilayet or Sandjak (respectively) of:										
Adrianople	2	300	3	—	—	—	—	—	—	—
Angora	6	—	—	—	—	80	7468	350	4	50
Sivas	65	—	—	—	—	—	—	5500	8	—
Kastamui	4	—	—	—	—	125	8000	—	—	—
Boli	15	6200	1	—	—	308	13068	2000	1	50
Karassul	25	—	—	—	—	67	13000	1000	3	—
Dardanelles	4	—	—	—	—	—	—	500	5	—
Mentcher	37	—	—	—	—	—	—	—	—	—
Diarbekir	3	—	—	—	—	—	—	120	—	—
Trebizond	—	—	—	—	—	—	—	150	3	—
Mosul	75	1000	2	—	—	—	—	700	15	—
Marash	1	—	—	—	—	—	—	1000	5	—
Syria	9	—	—	—	—	30	—	350	2	50
total:	11979	—	—	—	228	300	—	—	—	—

[1]) 1 dönum = 919 sq. m. — [2]) 1 oka = 1282 grams. — [3]) 1 piaster (= 100 centimes) = about 2½ d. — [4]) 1 dirhöm = 3,207 grams (400 dirhöm = 1 oka).

As may be deduced from table II, rose growing in Anatolia has its greatest extent in the two sandjaks of Burdur and Isparta (Sparta), situated in the western part of the vilayet of Konieh, especially in the neighbourhood of the two capitals of the same name as the sandjaks. Here, as everywhere in Anatolia, it is exclusively the native population who occupy themselves with this cultivation, and by no means the Turks who immigrated from Bulgaria. The Government is trying to extend it still further and has published a little illustrated manual for the instruction of the peasants: "The Book on Planning, Planting and Care of Rose Gardens and on the Obtention of Otto of Roses" (vol. 9 of the Library of the Ministry of Commerce and Agriculture. Constantinople, Library "Kadar", 1328 [1912]). In many places of the country further experiments of cultivation are made, for which the Government supplies the slips free of cost. Some of them have had to be given up as hopeless, *e. g.* near Tire, Ödemish, Kassaba, Ushak and, quite recently, near Konieh. Others, on the contrary, are most promising, the fields near Mughla developing especially well, where there is every prospect of a flourishing rose oil industry. Two years ago, a start was made there with 40 dönums and it is planned to increase the area next year to 400 dönums.

One of the principal conditions for the prosperity of the roses and for obtaining a good crop with a satisfactory yield of oil is a mild climate with a temperature oscillating as little as possible and with sufficient rainfall. Especially in the harvest time, dry weather is of the greatest disadvantage for the yield of oil. The most appropriate kind of soil is sandy clay with a sufficient proportion of lime and humus in it; it must be deep and keep the moisture well, without being too damp. Hard, stony, and especially also damp, soils with impermeable subsoil are altogether unsuitable, the same as low-lying valleys surrounded by mountains. The best site for the rose gardens is on free, gentle slopes in the direction towards north, north-east or north-west, so that they are protected against the warm South winds which diminish considerably the yield of flowers and especially that of oil.

All these conditions are most satisfactorily given in the present chief rose districts of Burdur and Isparta, in the former perhaps even better than in the latter, where a regular irrigation is required in summer. Before the war, a French company planned to use the available water power for an electricity works connected with a pumping station in order to water the fields. The said two districts and the sandjak of Mentcher (Mughla) offer, as far as the present experiments and experience go, the best conditions for rose growing and have every prospect of developing in the future on a still larger scale. Also in the vilayet of Brussa, where rose growing was introduced in 1909 and where there is now a special official, a fairly considerable extension has taken place in the last years. Of late, the peasants there even go so far as to destroy the mulberry plantations, owing to the present great depression in the silk industry, and replace them by roses. However, in Brussa the conditions do not seem to be favourable everywhere for rose growing, especially as the soil is too damp in some places, so that the peasants there run the risk of experiencing a great disappointment with their rose fields. In the vilayet of Aïdin (Smyrna), the conditions are unfavourable, generally speaking, as it is too hot and too dry during the flowering season. In consequence, rose growing has scarcely extended there in recent times; in the sandjak of Denislü, it only rose from 100 to 132 dönums in the last three years, and in the whole vilayet of Aïdin it comes now to about 400 dönums. It never had the extent of 5312 dönums, mentioned in the official statistics, this figure having only been arrived at by erroneously indicating 5000 dönums for the kasa of Bulladan district of Denislü, where there are in reality only 28 dönums of rose gardens. It is said that rose

growing has increased fairly well quite lately in the district of Diarbekir. It has gone down, however, in the sandjak of Afion-Karahissar, especially in the kasa of Afion-Karahissar, where only 5 dönums of the former 50 are left, the others having been destroyed by animals or been neglected, owing to the numerous calls to the colours and, above all, to the fact that most of the Armenians, formerly residing there, have been transferred some time ago.

The Anatolian peasants are by no means so opposed to innovations as is generally supposed; in fact, many of them started with ability growing roses, when the extraordinarily high prices, paid for Anatolian otto of roses a few years back, promised uncommonly good profits. But in many cases a discouragement set in, as the prices went down soon after, although they nevertheless still left a fair margin, for everybody had been spoilt by the former elevated quotations. The disappointment increased as, with the outbreak of war, the sales became difficult and the prices sank still further, so that at present there is on a whole but little inclination to extend the fields. It is even reported that in some places, where the poppies prosper, one intends to destroy part of the rose fields, should the present low prices continue (see farther ahead), and to grow the now remunerative poppies instead.

The greater part of the roses grown in Anatolia are said to descend from the shrubs smuggled in 22 years ago from Kazanlik. The variety with rosy double flowers, each with 35 to 40 petals, still grown near Kazanlik, predominates. About 350 to 400 flowers are required for 1 oka = 1282 grams. The white-flowering rose is grown on a smaller scale and almost only in the vilayet of Brussa. It yields more flowers but less oil, and this only fetches lower prices, but is willingly bought by the dealers, who mix it with the more valuable otto from the rosy Kazanlik flowers and thus do good business.

Besides, the indigenous so-called "hafis" rose is grown in the vilayet of Brussa. It has smaller leaves, no thorns and small, double, dark red flowers. Its yield in flowers and oil is extraordinarily high, but the latter is said to be inferior. However, the dealers buy it willingly for the reason stated above. The Brussa peasants are specially inclined to grow this "hafis" rose, slips of which have also been sent to Bulgaria. In Burdur, a start was made to plant this kind, but the Government ordered the shrubs to be removed, prompted by the justified desire to prevent the quality of the rose oils from getting inferior, so that at present the "hafis" and the white Kazanlik rose are only grown in the vilayet of Brussa.

Rose growing in Asia Minor is done exclusively on a small scale. In most cases the rose gardens only comprise a few dönums. Near Burdur, they are surrounded by brick walls, which are in bad repair, however, since the great earthquake of 1913. It is rumoured that then 400 dönums of rose gardens sank to the bottom of the Burdur Lake and are now about $6^1/_2$ metres below its level.

The care of the gardens reduces itself to keeping the interstices between the rows of rose bushes free of weeds and to ploughing or hoeing the soil or, at least, loosening it round the shrubs. In the often rainless time from June to October, the gardens must be watered at intervals, for which purpose small water furrows are made round the shrubs. In November, the soil is heaped up about 10 cm. high round the roots, in order to protect them against frost. In March, the dry or diseased branches are removed and every 10 years the shrubs are cut down almost to the ground. It is advantageous to manure every 3 or 4 years.

The gardens give the first crop in the second year after having been laid out; it goes on rising in the second and third, reaching its climax in the fourth, following

years. In a favourable situation, on good soil, and if a little care is bestowed on the shrubs, these may last 20 to 25 years, when they must be replaced by new ones.

The Anatolian rose fields have, it is true, not much to suffer from parasites, however, if they are not treated properly, some diseases are to be noticed. The above mentioned "hafis" rose is said to excel by special hardiness, as it is not attacked by any disease so to speak. The most disagreeable disease of the red Kazanlik rose is due to a fungus attacking the roots and caused by excessive watering. Another disagreeable parasite, it is said, is a green worm (most likely a grub living in the soil) which gnaws the roots; its occurrence is favoured by warm soils, so that it is better not to use horse manure, but cow manure. Mineral fertilizers are unknown. Sometimes, the rose buds are destroyed by a beetle and the leaves eaten by caterpillars. Further-

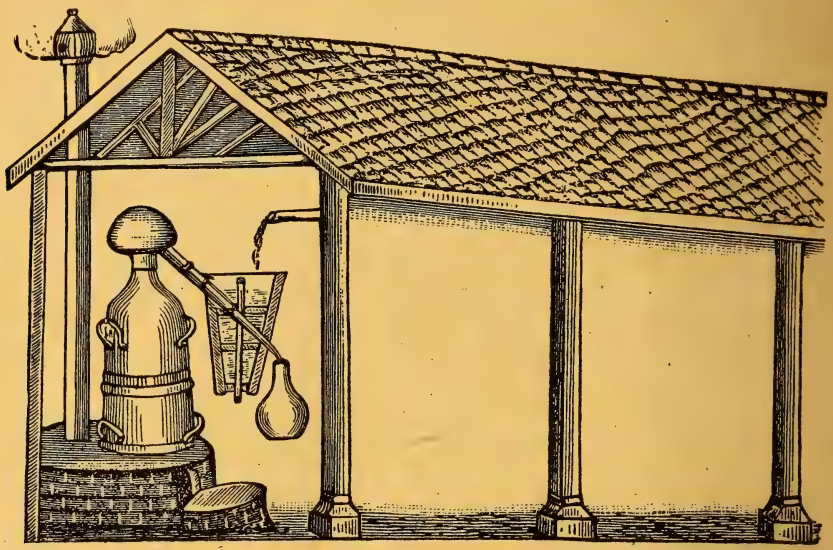
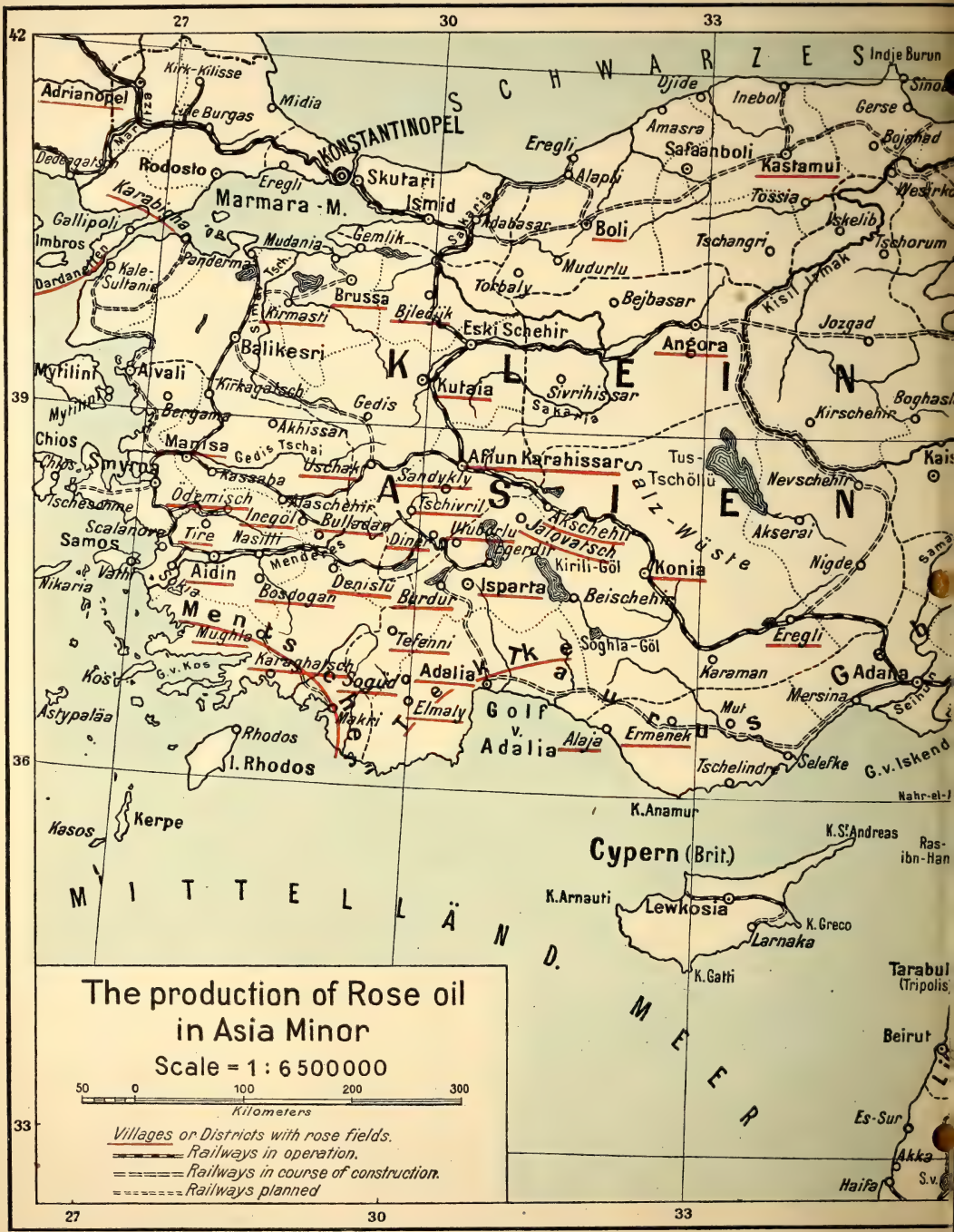


Fig. 1. Simple distilling plant (as per official "Manual").

more, the locusts, which in the last two years devastated quite specially the chief rose districts of Burdur and Isparta, have played havoc with the rose fields. But the Government has, last year, created an organisation under German management and on a large scale in order to combat this plague, so that it will probably be deprived of its threatening character within 2 or 3 years[1]).

The harvest takes place from the middle of May up to the middle of June. The yield depends largely upon the weather. As per verbal communications made to me by rose farmers in the main districts, it oscillates with the red Kazanlik roses between 100 and 300 okas (1 oka = 1282 grams) per dönum, *i. e.* between 1400 and 4200 kilos per hectare. According to the official book of instructions previously mentioned, the yields of red Kazanlik roses ought to be of 150 okas in the first, up to 500 in the second and afterwards up to 800 per dönum, which would correspond to 2000, 7000 and 11000 kilos per hectare; the latter figure appears to be rather largely exaggerated. On an average,

[1]) Comp. the author's treatise now under press in the *Umschau* and the *Zeitschrift für angewandte Entomologie.*


The production of Rose oil
in Asia Minor
Scale = 1 : 6 500000
50 0 100 200 300
Kilometers
Villages or Districts with rose fields.
Railways in operation.
Railways in course of construction.
Railways planned
SCHWARZES MEER
MITTELLÄNDISCHES MEER
KONSTANTINOPEL
Adrianopel
Midia
Kirk-Kilisse
Lule Burgas
Rodosto
Eregli
Skutari
Ismid
Marmara-M.
Gallipoli
Imbros
Dardanellen
Kale-Sultanie
Mudania
Gemlik
Brussa
Kirmasti
Balikesri
Myrilini
Alvali
Bergama
Kirkagatsch
Gedis
Akhissar
Manisa
Chios
Smyrna
Kassaba
Tschesmme
Odemisch
Inegöl
Scalanova
Samos
Tire
Nasilli
Aidin
Vathi
Nikaria
Bulladan
Dinar
Denisli
Bosdogan
Mughla
Karaghatsch
Kos
Astypaläa
Rhodos
I. Rhodos
Kasos
Kerpe
Boli
Tschangri
Mudurlu
Tokbaly
Bejbasar
Eski Schehir
Kutaia
Sivrihissar
Afiun Karahissar
Tschischvril
Sandykly
Uschak
Jalovatsch
Egerdir
Kirili-Göl
Isparta
Beischehir
Bardur
Tefenni
Soghla-Göl
Adalia
Elmaly
Golf v. Adalia
Alaja
Ermenek
Konia
Akserai
Nevschehir
Tus-Tschöllü
Salz-Wüste
Karaman
Eregli
Mersina
Adana
Tschelindre
Selefke
G.v. Iskend
K. Anamur
Cypern (Brit.)
K. S. Andreas
K. Arnauti
Lewkosia
K. Greco
Larnaka
K. Gatti
Tarabul (Tripolis)
Beirut
Es-Sur
Akka
Haifa
Kisil Irmak
Jozgad
Angora
Kirschehir
Boghasl
Kais
Nigde
Amasra
Ineboli
Djide
Indje Burun
Gerse
Kastamui
Tossia
Iskelib
Tschorum
Safaanboli
Alapli
Ada-basar
Sakaria

39
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45
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33
Kutais
Gori
Tiflis
Signach
Pori
Batum
Achalzich
Trans-Kaukasien
Kura
Samsun
Tscharschembe
Atina
Trapezunt
Ordu
Ünie
Kiresün
Ardahan
Alexandropol
Tschoruk
Olty
Kars
Nowo Bajaset
Gökscha S.
Gümischchane
Baiburt
Kagysman
Eriwan
Kara Hissar
Tokat
Kelkit Tschai
Erserum
ARMENIEN
Bajasid
Aras
Dawalu
Sivas
Ersingjan
Divrik
Kara su (West. Euphrat)
Egin
Chosat
Euphrat
Melasgirt
Nachitschewan
Munad-Su (Öst.
Arabkir
Choi
Musch
Wan S.
Wan
Charput
Malatia
Albistan
Arghana
Bitlis
Schattakh
Urmia S.
Sert
Diarbekr
Djulamerk
Seitun
Besni
Sewerek
Tigris
Midjat
Djesiret-ibn-Omar
Urmia
Marasch
Amadije
Mardin
Ainlab
Birdejik
Urfa
Feischabur
Killäz
Rewandus
Membidj
Mosul
Alexandrette
Aleppo
(Haleb)
Erbil
Antiochia
Ragga
Meskene
Euphrat
Altin Köprü
Kerkuk
Es-Saan
Ed-Deir
Chabur
Hama
Zori
Mejadin
Tigris
Homs
Sichne
Chanikin
Hermül
Anah
Baalbek
Badiet-esch-Scham
Euphrat
BAGHDAD
Damascus
Kebessa
Syrische Wüste
Kerbela
MESOPOTAMIEN
39 Östl. L.v. Greenw.
42
45

The production of Rose oil in Asia Minor
Scale = 1 : 6 500 000
50 0 100 200 300
Kilometers
Villages or Districts with rose fields.
Railways in operation.
Railways in course of construction.
Railways planned
SCHWARZES MEER
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Marmara-M.
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Rodosto
Skutari
Ismid
Brussa
Balikesri
Eski Schehir
Angora
Kutaia
Afiun Karahissar
Konia
Isparta
Adalia
Golf v. Adalia
Smyrna
Aidin
Mendos
Samos
Chios
Mytilini
Alvali
Bergama
Manisa
Ak-Denis
Scalanova
Rhodos
I. Rhodos
Kos
Kerpe
Cypern (Brit.)
Lewkosia
Larnaka
K. Greco
K. Anamur
K. St. Andreas
Ras-ibn-Hani
Ladikije
Tarabulus (Tripolis)
Beirut
Damascus
Es-Sur
Akka
Haifa
Hama
Homs
Aleppo (Haleb)
Antiochia
Alexandrette
Meskene
Aintab
Birdejik
Urfa
Marasch
Adana
Mersina
Tschelindre
Selefke
Ermenek
Alaja
Karaman
Eregli
Nigde
Kaisarie
Nevschehir
Kirschehir
Boghaslajan
Sivas
Tokat
Amasia
Tschorum
Iskelib
Tossia
Kastamuni
Samsun
Sinope
Indje Burun
Trapezunt
Ordu
Kiresün
Ersingjan
Erserum
Kara Hissar
Divrik
Arabkir
Malatia
Charput
Diarbekr
Mardin
Midjat
Sewerek
Besni
Seitun
Nadjin
Albistan
Sis
Tschorum
Amasra
Safranboli
Eregli
Ineboli
Gerse
Boiabad
Ujide
Midia
Kirk-Kilisse
Lule Burgas
Tschorlu
Tschanghri
Jozgad
Bejbasar
Mudurlu
Tschobaly
Biledjik
Sivrihissar
Gedis
Kutahia
Tschivril
Sandykly
Uschak
Akserai
Konia
Beischehir
Bardur
Denisli
Bosdogan
Tefenni
Soghla-Göl
Elmaly
Ermenek
Mut
Mersina

one may perhaps safely count upon a yield of 300 okas per dönum or 4200 kilos per hectare. In 1916, the crop was passable, but the yield in oil especially bad, owing to the drought. Whilst in favourable weather 10 kilos of red Kazanlik roses yield 1 miskal = 4.811 grams of oil, up to 20 kilos are required in bad years. White Kazanlik roses are supposed

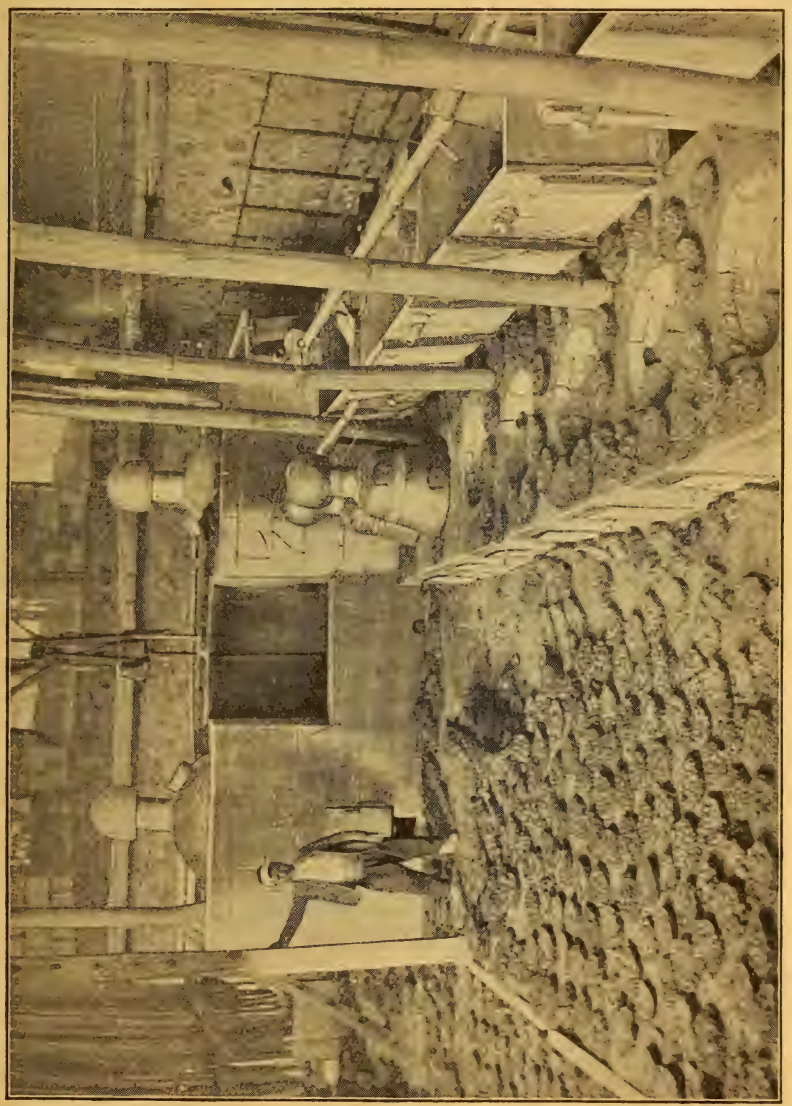

Fig. 2. Rose oil factory in Isparta.

to yield in favourable years up to 1000 okas of flowers per dönum, *i. e.* about 11000 kilos per hectare. As an average yield, 350 okas per dönum was indicated to me at Brussa, for white Kazanlik roses. In favourable years, 1 miskal of oil is obtained from 18 kilos of flowers, whereas in bad ones about 23 kilos are required. The yield of the indigenous "hafis" rose, cultivated in certain parts of the vilayet of Brussa, is said to arrive

even at 1000 to 1500 okas per dönum or 14000 to 21000 kilos per hectare, figures which hardly seem probable. In Brussa, one counts on an average yield, for this species, of 400 okas per dönum, equal to about 5600 kilos per hectare. The yield in oil of the "hafis" roses exceeds that of the red Kazanlik roses: for 1 miskal of oil, only 8 okas of "hafis" petals are required in ordinary years[1]).

The flowers are used exclusively for making otto of roses and rose water, the manufacture of rose extract (with the aid of light petroleum) being unknown. The farmers do not always distil their own roses, but frequently sell the whole crop to distillers. Distilling is carried on in technically very backward plants, of which there are a fair number. It is said that in the little place of Isparta and its immediate neighbourhood there are 75 distilleries; in Burdur and surroundings, 40 to 50 distilleries are working during the "season" and in the vilayet of Brussa, there are 35 distilling plants and one "factory".

The distilling apparatus are made in the country itself. Most of them are of a medieval kind, and even the larger works are far from being somewhat up to date.

Fig. 3.
Glass receiver for the rose water (as per official "Manual").

All the distilling apparatus are made for wood fires, but only in the vilayet of Brussa the large forests of the Olympus supply plenty of firewood. In the chief rose dictricts of Burdur and Isparta, however, which are very scantily wooded as it is, the production of rose oil, taking into consideration the well-known indifference of the authorities and the population, means utter deforestation of the district, for during the distilling period considerable quantities of firewood are used, the fittest being that of pines, beeches, or willows. Already now there is quite a pronounced fire wood calamity there and the extraordinarily high prices, which, it is true, have certainly been caused in part by speculation, raise the costs of distillation in quite an abnormal way. The Government recommends, and encourages the farmers, to plant the fast growing willows, in order to meet this trouble as soon as possible, but the fuel question will nevertheless continue to be one of the most important for the whole rose industry and we shall have to deal with it specially later on.

Fig. 4.
Glass receiver to hold the concentrated rose water for separating the oil off (as per official "Manual").

One of the largest factories in Isparta, which I visited, is shown on the photograph (fig. 2). It contains 10 small and 2 larger stills, placed in one row on brick fire-places. All the stills are made of tinned copper. The condensation is as simple as possible: open wooden boxes, arranged for running water, and through which the short, straight tin condensing tube goes. The two large stills, put up only recently, were shown as a modern installation. They are so arranged as to permit of withdrawing the whole distillation residue through the lateral opening on to a sieve, on which the extracted rose petals remain, whereas the hot liquid flows into a walled-in pit, from which it

[1]) In Bulgaria, it is said, one reckons as the yield of 1 hectare only 1400 to 2100 kilos of flowers. For the production of 1 miskal of otto of roses, in Kazanlik and Shipka, 17 kilos of white flowers or 12 kilos of red ones were necessary in 1914, which corresponds with the best years; in Brezovo, 10 to 11 kilos were required. The Prince of Sayn Wittgenstein indicates about 15 kilos of rose petals for 1 miskal of oil (comp. *Report* October **1916**, 48).

may be pumped up at once again into the still, in order to be used for a fresh distillation. The purpose of this arrangement is to use the liquid again and again, not so much for getting the last traces of oil out of it, but in order to save the expensive firewood. The solid residue, composed of the extracted petals, is now everywhere pressed into peculiar round cakes, which are dried and then serve the population as fuel for boiling and heating purposes. On our picture, which was taken after the "season" was over, we see these cakes, which remind one of the cakes made of camel excrements used in the scantily wooded parts of Anatolia, laid out for drying in the factory.

In the small stills shown on the picture (fig. 2), 60 okas of water are distilled with 6 okas of rose petals. The receivers used are large glass vessels with rather wide necks, holding about 6 okas. One distils enough to fill two such vessels, the first being the so-called good or "top-bottle"; the second, the bad or "foot-bottle". The contents of five "top" and five "foot" bottles, *i. e.* together 60 okas of simple rose-water, is put back into a still, previously cleaned, and from which a quantity is distilled off, sufficient to fill consecutively a flask holding 5 okas (I) and the two holding 6 okas each (II and III).

The residue in the still is not thrown away but used for distilling further quantities of flowers, after having been diluted with 17 okas of water.

The distillate III is mostly sold as rose-water, whereas the distillate II is distilled again, in the way just described, together with the contents of 9 "top" and "foot" bottles. In order to distinguish the bottles, different numbers of blue and of white glass-beads are tied round their necks, such as in the country the animals frequently wear as ornament or talisman.

The highly concentrated rose-water (I) is left in said flask to separate off the oil, which is then carefully removed with thin glass-tubes or small scoops. Owing to the ungainly shape of the vessels, considerable losses are unavoidable. A fair amount of the oil sticks to the upper curve of the flask and the great width of the necks allows but a thin layer of oil to form, which it is difficult to take off completely. The concentrated rose-water, from which the oil has been removed, is diluted with 9 bottles of simple rose-water, and the whole quantity concentrated again, as described before, to fill 3 flasks.

In Isparta, there is a kind of buying society of rose oil manufacturers, which controls the sales and advances money to the farmers as well as to the distillers or grants loans on rose oil. In Burdur, there is no such or similar institution. The Agricultural Bank in Constantinople, which is under state control, likewise grants loans or advances on rose oil.

The rose-water is consumed exclusively in the country itself and finds extensive use in religious ceremonies, for cosmetical purposes, in confectionery, for sweet dishes and so on[1]).

Most of the rose oil was hitherto supplied to France, the French Mikado Perfumery being named as the principal customer. The sales were managed specially by two Constantinople firms. Since the outbreak of the war, business is almost at a standstill. At the beginning of July 1916, about 100000 miskals of rose oil were stored in Isparta and 70000 miskals in Burdur, *i. e.* together more than 800 kilos of unsold rose oil.

The prices were rather high in comparison with what was paid for Bulgarian otto of roses before the war. The maximum paid in Burdur was 80 piasters per miskal,

[1]) It may also be mentioned that a certain amount of rose petals is used for making the highly valued rose petal jam, or rather was used, as at present the sugar is too dear for the purpose.

or in round figures about 165 Turkish pounds per kilo[1]). A short time before the war, it is said to have been 2500 francs per kilo. In July 1916, it was possible to buy at Burdur in the retail trade at 20 piasters per miskal, corresponding to 415 piasters per kilo. In Isparta, I was offered rose oil, in July 1916, at 1000 francs per kilo; in Brussa, in December 1916, the product of Kazanlik roses at 25 piasters per miskal, that of "hafis" roses at 20 piasters, and that of white roses at 40 piasters. Owing to the low prices, many natives then bought 1000 and more miskals each, in order to store these stocks for better times coming. May they be satisfied with their speculation!

The high prices, obtained so far for Anatolian rose oil, as compared with the Bulgarian product, are chiefly due to the fact that adulterations were happily almost unknown with it, whereas they flourish in Bulgaria. But in recent times, pedlars have tried, in Anatolia, to introduce adulterants to the farmers, *e. g.* oil of geranium, which was offered at from 300 to 400 piastres per oka and of which once 10 000 miskals were confiscated by the inspector of the rose industry, a short while ago. It has been motioned to prohibit officially the import of all such oils as are used or may be suitable for faking otto of roses.

In spite of the still existing "purity" of the Anatolian rose oils, which it is to be hoped will survive in future, and of their excellent quality, their price seems comparatively too high, especially if the production be further increased, in order to compete successfully with the rose oils of other origin. Exaggerated prices, besides, tend to induce consumers to use cheaper synthetic substitutes, which would diminish the possibilities of selling the genuine otto of roses. It is interesting, therefore, to investigate how high the costs of production are and at what prices the farmers are able to produce under normal and healthy conditions.

Let us suppose that 1 dönum yields on an average 300 okas of red Kazanlik roses, for which, in normal times, 1 to 1½ piastre was paid per oka. Sometimes, the price went up to 2 or even 3 piasters. It is remarkable that there is no normal price, *i. e.* that the price never depends upon the crop and on the quality of the petals, but is always fixed after the distillation is over and the oil sold in accordance with the price obtained for the oil. Last year, it was only ¼ piaster per oka in Isparta, 1 piaster per 3 okas in Burdur, and in Uluburlu even as low as ⅛ piaster per oka, owing to bad trade and in spite of the scanty crop.

The yield in petals, under normal conditions, represents an average value of 300 to 450 piastres per dönum or 3270 to 4910 piasters per hectare. Taking into consideration the comparatively low expenses for laying out the rose gardens and keeping them in order, the profit is rather high and the farmers may well be satisfied with it. It is acceptable even if the crop is inferior or if the price sinks to ½ piaster per oka of petals[2]).

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[1]) The former chaos of currency: gold, silver, bad piastres, and so on, has fortunalely been replaced by the decimal system according to the financial Act of April 1916: 1 Turkish pound = 100 piastres. 1 piastre has 40 paras. The rate of exchange in times of peace was about 18 s. 2 d.

[2]) For the sake of comparison, the returns of some other branches of agriculture are given hereafter, as per official statistics for the sandjak of Denislu: —

wheat: . . . 1329 (1913) from 25000 dönums = 120000 killehs (of 22 okas), price per killeh = 20 piasters
1330 (1914) „ 45000 „ = 130000 „ „ „ „ = 28 „
i. e. on an average per dönum 3.6 killehs at 24 piasters = 86.4 piasters.

barley: . . . 1329 from 26250 dönums = 208000 killehs (of 18 okas), price per killeh = 10 piasters
1330 „ 36000 „ = 215000 „ „ „ „ = 12 „
i. e. on an average per dönum 7 killehs at 11 piasters = 77 piasters.

From 8 to 15 okas of petals of the red Kazanlik rose are necessary to produce 1 miskal of oil, or 1670 to 3120 okas for 1 kilo. As per informations supplied by some rose oil dealers and manufacturers, the expenses with the present method of distilling, including interests, sinking-fund and keeping the rather primitive apparatus in good repair come to about 5 to 7 1/2 piasters per miskal, representing therefore the considerable amount of 1040 to 1560 piasters per kilo. The net costs of production for 1 kilo of rose oil on the spot would therefore have to be calculated approximately as follows: —

1670 to 3120 okas of petals at 1 to 1 1/2 piaster = 1670 to 3745 piasters
expenses for production . . . . . = 1040 „ 1560 „
together 2710 to 5305 piasters.

We will here leave apart the accessory profit made through the sale of the rose water. The prices named tally fairly well with the indications given me by Burdur rose oil merchants, who may, however, quite naturally have estimated rather high, according to which it would be possible to produce 1 miskal of rose oil for 16 piastres (*i. e.* 1 kilo for 3330 piasters) under normal conditions and with the present methods of manufacture.

As is to be seen, the "manufacturing expenses" come up to nearly 40 per cent. of the initial cost of the oil and are largely due to the expensive fire-wood. They could be reduced very considerably through modern apparatus for steam distillation and heating with coal, which would likewise increase the yield in oil. Coals could be procured easily and cheaply, especially for the present chief rose district Burdur and Isparta, as there are important coal-beds in the neighbourhood, along the Aïdin railway line. In several other places of the country, there are also coal-mines, which are being busily worked now, as imported coal is scarcely to be had any more. The coal, a kind of lignite, is of indifferent quality, it is true, but during the war it has found extensive use in the country, even for railways and steamers. Care being taken to improve the combustion by special ventilation appliances, one now uses it likewise with good results for locomobiles, the grates of which, however, suffer greatly from the sulphur compounds and, besides, the fuel consumption is increased. The destructive influence in the grates would scarcely matter, as the yearly campaign lasts but

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Indian corn: 1329 from 10000 dönums = 70400 killehs (of 18 okas), price per killeh = 12 piasters
1330 „ 11000 „ = 81200 „ „ „ „ = 15 „
i. e. on an average per dönum 6.8 killehs at 13.5 piasters = **91.8** piasters.

poppies: . . 1329 from 180 dönums = { 5400 okas of seeds . . . . price per oka = 1.5 piaster
+ 360 „ „ opium . . . . „ „ „ = 220 piasters

1330 „ 150 „ = { 4500 „ „ seeds . . . . „ „ „ = 1.5 piaster
+ 260 „ „ opium . . . . „ „ „ = 160 piasters

i. e. on an average per dönum 30 okas of seeds at 1.5 piaster = 45 piasters
+ 1.865 oka of opium „ 190 piasters = 354 „
together = **399** piasters

sesam: . . . 1329 from 4000 dönums = 270000 okas at 2 piasters
1330 „ 11000 „ = 310000 „ „ 2 „
i. e. on an average per dönum 38.6 okas at 2 piasters = **77.2** piasters.

According to the "Agricultural Statistics for the year 1329", published by the General Governing Board for Statistics of the Ministry of Commerce and Agriculture, the average crops in Turkey in 1329 were as follows per dönum: —

wheat . . . . 4.4 killehs at 21.15 piasters = 93.6 piasters,
barley . . . . 6.0 „ „ 12.42 „ = 74.5 „ ,
Indian corn . . 5.3 „ „ 13.80 „ = 73.1 „ ,
sesam . . . . 36.7 „ „ 2.17 „ = 79.6 „ .

four weeks. Otherwise it is only a matter of calculation, whether to choose the expensive imported coal or the cheaper indigenous product, but in any case no lack of coal is to be dreaded even in the case of political troubles, owing to the presence of coal-mines in the country.

The manufactures of otto of roses and the Government show a keen interest for the introduction of new distilling apparatus and have in several cases granted considerable amounts for the purpose.

It is easily understood that modern distilling plants will only prove remunerative in the large rose-growing centres, where the rose gardens are not too far apart from one another. It would naturally be the best for all rose growers to create such plants on a co-operative basis, but, generally, they are quite small farmers, who own no more than a few dönums and have neither capital nor initiative for such undertakings on a large scale. Only the more important, well-funded people or private companies may come into consideration. German capital and German enterprise, combined with a sound knowledge of the matter, would here find a promising field of action, especially in connection with rose farming, supposing that those concerned understand how to adapt themselves to the peculiarities of the country and of its people, with whom it is often difficult to deal.

Constantinople, December 19th, 1916.

Following up above article, we would here insert a communication of the German Consulate in Constantinople[1]), dated January 6th, 1917, according to which it is impossible to give figures with a claim to exactitude as to the production of rose oil in 1916, the reports from the interior being altogether inadequate[2]). The yield is estimated at from 40000 to 50000 miskals (1 miskal = 4.811 grams). Owing to the abundance of flowers and the favourable weather, a good result was counted upon, but as the roses contained but little oil and there was a lack of hands, the yield came far short of that in the previous year. Notwithstanding, the olfactive value of the 1916 oil is very high.

Like in 1915, there was a deadlock in the rose trade in the past year, there being almost no demand at all. The low level, the value of the article had reached, attracted the speculation, which sent the prices up to 22 and even 25 piastres per miskal. These speculative purchases had, however, little to do with the export. In consequence, the crops of the two last years will therefore almost entirely be in Turkey still, and it is thought that the present stocks are comparatively speaking rather considerable.

Rose Oil, Bulgarian. As per a report of the German Consulate in Sofia, dated May 24th, 1917[3]), the rose buds had appeared towards the end of April almost everywhere in Kazanlik, Karlovo, Nova Zagora, &c. Scarcely any damage has been caused by cold weather or frost, but a kind of small grub, which appeared this year in larger numbers than before, handicapped many of the young flower-buds in their development. Hot and dry weather during the crop most unfavourably influenced the yield in oil, as is generally the case, so that according to the investigations of our informant, there were 4000 kilos of flowers required, this year, to produce 1 kilo of oil. The total yield of the 1917 crop is estimated at 2100 kilos, which means a further shortage,

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[1]) *Nachr. f. Hand., Ind. u. Landw.* **1917**, N° 8, page 3. — [2]) *Asia Minor* (Anatolia). — [3]) *Nachr. f. Hand., Ind. u. Landw.* **1917**, N° 46, page 2.

as compared with the 2800 kilos of 1916 and 3400 kilos of 1915. The great demand for grains, caused through the war, naturally diverted the interest of the Bulgarian farmers from the rose fields, many hectares of which are said to have been converted into corn-fields, as these offer better chances. It is true that the present condition of the rose oil market is not very encouraging, the export having sunk to 2100 kilos in 1915 and as low as 1600 kilos in 1916, against 3200 in 1914 and 5500 in 1913. The producers of rose oil are reported to have lost a fair amount of money with the prices of the last years, as the sales were slow, there being no inclination for speculative undertakings, and living became more expensive from week to week. Recently, nearly all Bulgarian landowners have become rich through the trade with cereals, tobacco, beans, and potatoes, so that they are in a position to reserve their stocks of rose oil for better times. They not only succeeded in preventing a further fall but in raising the market price by about 1000 marks, so that 2200 marks or so are asked for 1 kilo of the 1917 crop. Large quantities — the figure mentioned is about 3500 kilos — of the crops from 1914 up to 1916 are said to be in their hands, while merchants, banks and other middlemen hold a further 2500 kilos. Cheap offers from the latter circles indicate from the very beginning that the article is "faked" a good deal, a fact which was demonstrated by the results of various examinations.

According to the *Deutsche Balkan-Zeitung* of June 25th, 1917, the association of rose oil producers in Kazanlik published an appeal asking all their fellow-manufactures in the kingdom to unite and organize themselves. The purpose is to exclude all unfair competition and to supply only pure genuine rose oil, thus regulating the price at the same time. The association is said to have bought up the whole last year's crop (?) and to be in negotiations about this year's. After the previous failures, we feel inclined to doubt that it will be possible to "gather all rose oil producers around one flag" and to reach the goal of thoroughly abolishing all unfair dealings in the rose oil industry, striven after in vain for so many years.

Rosemary Oil. As owing to the war no supplies are available from Austria-Hungary, it seems that the interest for distilling oil of rosemary revives in France. So there is an article by H. Blin[1]), entitled *La distillation du romarin en Roussillon*, in which the production of said oil in this most southern part of France is discussed. Rosemary grows in masses on the chalky soil of Roussillon, Provence and Languedoc, and it further occurs in the mountains, such as the south-eastern Pyrenees, the Basses-Alpes, in the Gard, Drôme, Var, Alpes-Maritimes, Vaucluse departments, Mont Ventoux, on the Montagne de Lure, in the Lubéron, the Dauphiné, the Cevennes, Tunis, Spain, Corsica, Sicily, &c.

Rosemary is very sensitive to cold, so that frosts are nearly always disastrous. In the south-eastern departments it is often destroyed by flocks of sheep. According to Blin it would not be sufficient to forbid pasturing on the tracts of land where rosemary grows, but the cultivation of this plant ought to be encouraged. Rosemary thrives on dry, pervious soil in sunny places and is propagated in autumn by means of slips, cuttings or suckers, or in March and April by means of seeds. After two or three years it may be cut, the plants being ready for distillation as soon as the flowers have opened and before they are dried up. Rosemary flowers from beginning of June into September. The Roussillon pickers generally are paid 1 to 1½ franc per 100 kilos.

All parts of the plant are distilled, except the woody stalk. The leaves supply the best oil.

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[1]) *Parfumerie moderne* **10** (1917), 24, 44.

On the mountains, the distillation is carried out on the spot in portable stills, which are put up in the vicinity of a brook. Where there are fairly good paths, all the material of the neighbourhood is united and distilled in larger installations. In France as well as on the Balearic Islands, in Turkey, on Crete, &c., one has alway slimited oneself to distilling the wild-growing plant, but with a view of successfully meeting the Dalmatian competition, it is absolutely necessary to grow rosemary.

The small distillers on the mountains use very antiquated stills. In the eastern Pyrenees, the better-equipped distilleries are provided with copper stills, holding from 1200 to 1500 kilos and adapted for water and steam distillation. When distilling with water, the stills are filled with it to the height of about 30 cm., and the distillation takes 3 hours or so. Distillation with steam requires only $1^1/_2$ hours. The stills are discharged by removing the exhausted material together with the sieve bottom, the former being dried in the sun and then used as fuel.

For some time past, the smaller Roussillon distillers seem to show more interest for small portable stills of modern construction, which hold about 100 kilos of the fresh herb and used to cost 450 francs or so, before the war. The larger installations of the eastern Pyrenees are equipped with 6 or 8 stills, the steam being developed in a boiler. Eeach still does not contain more than 300 kilos of rosemary, but it is well distilled and the oil obtained is excellent. In Salces, near Perpignan, a factory works the herb of an area of about 6000 hectares, covered with rosemary, thus producing from 4000 to 5000 kilos of oil yearly. The steam is developed in a boiler with a heating-surface of from 30 to 35 sq.m., the exhausted rosemary being used as fuel. 14 workmen are employed, 8 in day-time and 6 over night. Every 24 hours, about 8000 kilos of rosemary are distilled, yielding 30 to 40 kilos of oil or 0.38 to 0.5 per cent., according to the season and the weather. The stills have a capacity of about 1500 litres and can hold up to 1100 kilos of herb. Besides, a more complicated process is in use, requiring two stills connected by means of a three-way-cock *(alambics mariés)*. After both stills have been filled, steam is let into one for about $1^1/_2$ hours, when the three-way-cock is turned so that the steam goes from the first still into the second. After an hour, the distillation is interrupted and the steam let direct into the second still. One distils for $1^1/_2$ hours and then conducts the steam into the first still, which has meanwhile been refilled with fresh material. In this way, there is an economy of steam and water, but much attention is required.

The small Roussillon distillers obtain about 1250 grams of oil from 1000 kilos of rosemary, corresponding to a yield of 0.125 per cent. Better results are obtained with modern apparatus, as mentioned before.

Sage Oil. Sage, *Salvia officinalis*, L.[1]), a labiate spread over the whole coast of the Mediterranean as far as Spain, grows on the mountains and hills of Croatia, Dalmatia and the islands of Veglia and Cherso, in the Quarnero Gulf. The plant, which seems to prefer lime-stone formations, is the only herb that grows on some of the hills near Fiume.

It is collected for exportation in Dalmatia and on the Adriatic islands. Trieste is the export centre, whereas Fiume exports but little.

The best kind of sage, it is stated, grows near Malinska, on the island of Veglia, where the surrounding district is known as the sage region. There it is gathered before blooming and by cutting the stem rather than pulling the plant up by the roots,

[1]) *Journ. Royal Soc. of Arts; Perfum. Record* 7 (1916), 369.

as is the practice in some parts. While wild sage seems very plentiful in the vicinity of Fiume, the business of gathering it commercially has not been taken up, except on the neighbouring islands of Cherso and Veglia. During its blooming season the bees gather the nectar, and genuine sage honey commands the highest price, owing to its flavour.

In the regions where sage grows, its leaves are boiled with vinegar and used as a tonic. A decoction is also made from it for darkening the hair. It is used more as a domestic remedy than by physicians. It is given in the form of a tea for night-sweating and diarrhœa, and is used as a gargle for bleeding gums, catarrh, and to prevent excessive flow of saliva. The oil is used for removing heavy collections of mucus in the respiratory organs. Its fresh leaves are used as a tooth wash, and it is a common ingredient in tooth powders. The dry leaves are used for smoking as a remedy for asthma. The domestic consumption accounts for a very small proportion of the crop, as it is gathered chiefly for export.

The leaves are harvested from May to September, but those plucked in midsummer are considered the best. The general opinion is that it should be gathered before the bloom opens, but the Austrian Pharmacopœia says it is best when gathered during bloom.

The only treatment required for the leaves intended for export is drying. The leaves must not be exposed to sunlight while being dried, hence drying is done in the cottages of the pickers. When thoroughly dried, the woolly hairy dust of the leaves is removed by sifting. It is estimated that 9 lbs. of fresh leaves yield 2 lbs. of the dried herb. After drying, the leaves are sorted and generally pressed into bales of 100 to 300 kilos (220 to 660 lbs.) and bound with iron bands.

The essential oil is obtained by steam distillation of the fresh leaves, producing from 1.3 to 2.5 per cent. of oil.

Large quantities of sage were formerly sent to Germany, whence a good deal was shipped to Great Britain and the United States.

Sandalwood Oil, East Indian. By far the largest part of the abundant stocks of the German distillate still available is in our hands. We were thus in a position to counteract successfully every attempt to push the prices.

There are no recent news as to the fate of the newly erected factory of the Mysore Government[1]), so that one is entitled to suppose that it did not get much farther than the starting point, as otherwise the English would surely not have missed to announce to the world at large, with more or less fanciful embellishments, this "triumph" of English enterprise.

Some time ago, the German professional press copied on several occasions a rumour set afloat in England, according to which we had established a branch factory in Spain with a view to import sandalwood there and to do the French sandalwood oil business from Spain, thus preventing that this trade might be spoilt for us in the hostile countries. It was even added that we had offered the oil at 101 francs per kilo. This rumour is derived from the *Madras Weekly Mail* of September 22nd, 1916, which brings also several other news from India on sandalwood and its elaboration. We beg to state that the notice concerning our firm is a *lie* from beginning to end and refrain from reporting on the further contents of the article, as it is to be presumed that it will agree with the facts just as little. There they talk big about the Mysore distill-

[1]) *Report* October **1916**, 51.

ing plant already mentioned, but we can afford to wait and see what its capacity will be like in reality.

We have had some further experience in the adulterations that are frequently practiced with sandalwood oil. In one case benzyl alcohol had again been added, a substitute which seems to be much in vogue nowadays, as we have frequently observed it as an adulterant of this oil during the last years[1]). This sample showed quite similar qualities to those of the adulterated product mentioned in our last *Report* (p. 49), namely: $d_{15°}$ 0.9784; α_D — 11° 15′; acid value 2.7; ester value 7.5; ester value after acetylation 197.9; soluble in 4 vols. of 70 per cent. alcohol, the solution on further dilution turning opalescent or even turbid. In this case also, just like in the former one, a discrepancy was evident, *e. g.*, the rotation was too high in spite of a high acetylation figure, besides which the solubility was far from being satisfactory. Our suspicions that this was not the case of a pure sandalwood distillate were abundantly confirmed on closer examination. Notwithstanding smallness of the sample we succeeded in separating benzyl alcohol from it by fractionated distillation and identifying it by conversion into benzyl acetate. Benzyl alcohol being calculated as santalol in acetylation, the santalol content of 91.3 per cent., deduced from the above-mentioned acetylation value, only apparently corresponds to this figure, in reality it is much lower.

Perhaps this is not the only adulteration, as the addition of benzyl alcohol may explain the insufficient rotation, but not the bad solubility which on the contrary is increased considerably thereby; possibly of course the original oil may have been very inferior in quality.

Another sandal oil sample gave the following figures:— $d_{15°}$ 0.9561; α_D — 19° 48′; acid value 1.5; ester value 5.6; ester value after acetylation 61.6; soluble only in 7 vols. of 90 per cent. alcohol. The adulteration is proved in this case by the too low specific gravity, the much too low acetylation figure (normally not below 196), and the quite insufficient solubility. The boiling test was also unsatisfactory, as 70 per cent. distilled off already between 275 and 285°, whereas under similar conditions the first 70 per cent. of pure sandal oil distil between 285 and 305°.

From these observations and also from the quite different smell we must conclude that this product did not contain any East Indian sandal oil at all and that on the contrary it was a mixture of *cedar oil* and *West Indian sandal oil.*

Santalol, which determines the value of the East Indian oil, was totally absent in these 2 samples, but even if it had been present, the quantity would have been quite insufficient, as the acetylation figure would correspond to a santalol content of only 25.3 per cent., whereas a good East Indian product contains at least 90 per cent. thereof.

A third, also adulterated oil, was sent to us in capsules. Its specific gravity was too low ($d_{15°}$ 0.9691) and its ester value too high (26.3), which facts induced us to conclude that a fatty oil was present. Unfortunately it was impossible to determine the nature and quantity of the adulterant, as only 20 capsules were at our disposal.

All three oils had been sent to us for examination from Switzerland.

C. H. Briggs[2]) recommends the following method for determining the yield of volatile oil from small samples of sandalwood: 100 g. of the finely divided wood are extracted in a Soxhlet-apparatus with ether, the latter is evaporated on a water bath

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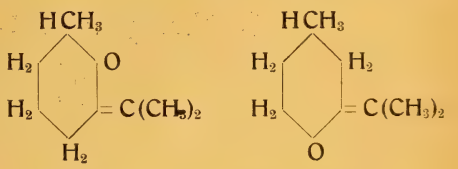
[1]) Comp. *Report* October **1913**, 98; October **1916**, 49, 50. — [2]) *Journ. ind. eng. Chemistry* 8 (1916), 428; *Journ. Soc. chem. Industry* **35** (1916), 706.

with the help of a current of air, and the residue, mixed with 35 cc. of glycerine, is distilled *in vacuo*, till 25 cc. of glycerine have passed over. The distillate is then mixed with 25 cc. of water, is agitated thrice with chloroform, using 25 cc. each time, and the extract then evaporated on the water bath in a tared dish, and finally the weight of the residue is determined after drying *in vacuo* over sulphuric acid. Twelve sandalwood samples from Mysore, treated in this manner, produced from 3.5 to 8.3 per cent. of oil. The oil, extracted by this process, showed a rotation of at least — 18° and was easily soluble at 25° in 5 vols. of 70 per cent. alcohol, whereas the oil obtained from the same wood by distillation required for solution more than 5 vols. of 70 per cent. alcohol and only showed a rotation of — 14° or even less. According to Briggs, these differences are caused by changes produced in the oil owing to prolonged contact with boiling water.

Unfortunately, we have not the original paper at our disposal, but only an abstract, hence our criticism must not be too severe. In any case Briggs' method seems to us quite unsuitable. Of course, two oils, produced by such different means, cannot possibly be compared, least of all, when proceeding in the arbitrary manner Briggs employs. Is the author really sure that all the components of the sandal oil have passed over with the 25 cc. glycerine distillate? Our sandalwood oils, obtained by distillation, were always easily soluble in 3 to 5 vols. of 70 per cent. alcohol and occasionally gave a rotation even over — 20° with a santalol content of about 95 per cent. Accordingly we are forced to conclude that Briggs hardly knows how to distil sandalwood in an appropriate manner.

Concerning an isomeric teresantalic acid, see page 154 of this *Report*.

Santolina Oil. L. Francesconi and N. Granata[1]) have continued their investigations on the structure of santolinenone which we have repeatedly discussed in our *Reports*[2]). They have discovered that β-santolinenone forms a liquid oxime and a simple hydroxylamine compound, melting between 63 and 64°. The oxalate of the hydroxylamine compound melts at 161°. Oxidation with mercuric oxide transforms the hydroxylamine compound into the pungent smelling nitroso-β-santolinenone melting between 60 and 62°. The best means to produce the hydroxylamine compound of β-santolinenone is to allow hydroxylamine hydrochloride in the presence of sodium bicarbonate act on an alcoholic-ethereal solution of the oil from *Santolina Chamæcyparis*. As yet it is impossible to determine exactly what the structure of β-santolinenone may be, possibly one of the two formulæ below may express it. Accordingly β-santolinenone is probably a derivative of *m*-cymene, hence the first known *m*-ketone found in a natural state.



Possible formulæ for β-santolinenone.

Sassafras Oil. A sassafras-wood oil sent to us for examination turned out to be ordinary safrol, as the following figures demonstrate: $d_{15°}$ 1.106; $\alpha_D \pm 0°$; $n_{D20°}$ 1.53648; freezing point + 10.6°; soluble in 3.5 vols. and more of 90 per cent. alcohol. Sassafras

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[1]) *Gazz. chim. ital.* **46** (1916), II. 251; *Chem. Zentralbl.* **1917**, I. 13. — [2]) Comp. *Report* April **1912**, 113; April **1915**, 41; October **1915**, 35.

oil is optically active and its specific gravity, refractive index, and freezing point are lower than the above-mentioned figures.

Although safrol is the main component of sassafras oil, it would be quite wrong to sell it as sassafras oil, as on the manufacturing scale it is made from cheap camphor oil and is accordingly much less expensive; to dispose of it as sassafras oil is flatly fraudulent.

This case again draws attention to the necessity of great care when buying volatile oils nowadays.

Spike Oil. Some years ago J. C. Umney[1]) put forward the demand that oil of spike should contain 30 to 40 per cent. of alcohols, calculated as linalol; oils with less were to be suspected as being adulterated with rosemary oil. He indicated the following constants for pure spike oil: $d_{15°}$ 0.905 to 0.915; α_D 0 to $+7°$; soluble in 1 to 3 vols. of 70 per cent. alcohol; alcohol content (determined by acetylation) over 30 per cent. Since then one has found that unadulterated spike oils also exist which are slightly lævorotatory (up to $-2°$).

Umney[2]) has now examined more thoroughly the amount of acetylisable compounds in spike oil. He divides spike oils into three classes: high grade ones with over 35 per cent. of alcohols, low grade ones containing 30 to 35 p. c., and adulerated ones with less than 30 per cent. He gives the following constants for a number of oils in form of a table, *viz.*,

For oils of the 1st class the specific gravity is 0.900 to 0.918; α_D -5 to $+4°5'$; the alcohol content 35.0 to 41.4 per cent.

Oils of the 2nd class have the specific gravity of 0.900 to 0.922; α_D -3 to $+6°$.

For adulterated oils (3rd class) Umney adds the following figures: specific gravity 0.897 to 0.921; α_D $-2°30'$ to $+12°30'$.

It is really hard to determine how far the data given by Umney may be used as a guide in the investigation of these oils; our opinion, however, is that the lower limits given for the specific gravity are rather too low. In the case of pure oils we have never found a lower value than 0.904, not even in oils which we have manufactured ourselves in the South of France from selected material. Accordingly, it appears to us that a specific gravity below 0.904 with simultaneous lævorotation and high alcohol content indicates the presence of lavender oil which can easily have been added by the plants worked up being mixed with lavender, as lavender and spike often grow close together. An admixture of lavandine, the hybrid between lavender and spike, is also imaginable. In any case Umney's data must be rigorously tested with warranted pure spike oil before they can obtain currency as standards.

Lavender flowers are generally used externally in the shape of herb-cushions and of infusions for the local treatment of rheumatism as slight irritants, whereas they are only rarely employed nowadays internally as stimulants and antispasmodics, for instance for the treatment of flatulence and colic. Quite by chance, namely in consequence of a change by mistake, Morpurgo[3]) ascertained that spike flowers (the blossoms of the large lavender, *Lavandula Spica*, D.C.), have a very pronounced diuretic effect. An infusion produced a strong polyuria which, however, disappeared again in a short time. As the urine did not show the presence of any abnormal

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[1]) *Chemist and Druggist* **52** (1898), 166; *Report* April **1898**, 48. — [2]) *Perfum. Record* **7** (1916), 239. — [3]) *Klinisch-therap. Wochenschr.* **1915**, 459; Merck's *Jahresbericht* **29** (1915), 288.

compounds during the action of the diuretic, experiments were made with it on patients suffering from defective discharge of urine, which lead to very good results; in fact, in almost all cases a notable increase of the urine discharged during 24 hours was observable. The treatment consisted of a dose of a hot infusion of 20 g. of spike flowers in 200 g. of water.

Summer Savory Oil. We may mention owing to its rarity a few particulars concerning a distillate of summer savory oil which we obtained, although the examination only included that of the sp. gravity and of the solubility. The colour was brown, it smelt of thyme and deposited crystals (paraffin?) already at ordinary temperature. The specific gravity was 0.9582 at 20°, the solubility was, apparently owing to the paraffin content, very bad, as even 10 vol. of 80 per cent. alcohol produced no clear solution. The oil-yield from the fresh herb was 0.09 per cent.

Tea Oil. Tea oil contains according to P. van Romburgh[1]) besides methyl salicylate a liquid boiling between 153 and 154° (740 mm.) and having a penetrating odour of fusel oil, reminding of the smell of tea. J. J. B. Deuss[2]) arrived at the same conclusion in the course of an examination of tea. This oil, obtained by distillation with the aid of steam, is yellow and has a strong smell of tea. On distillation *in vacuo* the oil is produced in a colourless state and can be divided by fractionation into two parts, of which the main portion boils at 156° and smells of tea, whereas the higher-boiling fraction passes over at 220° and consists of methyl salicylate.

Thyme Oil. Concerning the testing of the oil of thyme according to the German Pharmacopœia, 5th ed., *vide* page 86 of this *Report*.

Turpentine Oil. A most fraudulently adulterated oil of turpentine was sent us *via* Hamburg. The adulteration was proved not only by the smell, but particularly by the much too low specific gravity and also by its quite abnormal boiling characteristics, whereas the other constants showed nothing remarkable, with the only exception that the solubility was not all one could wish: $d_{15°}$ 0.832; α_D +3° 18′; acid value 1.9; ester value 7.5; soluble in about 10 vols. of 90 per cent. alcohol. Boiling characteristics, when distilled from a Ladenburg flask (745 mm.):—

1.	142 to 150°	6.0	per cent.
2.	150 „ 155°	9.4	„ „
3.	155 „ 160°	7.6	„ „
4.	160 „ 170°	30.0	„ „
5.	170 „ 180°	1.56	„ „
6.	180 „ 190°	9.4	„ „
7.	beyond 190°	22.0	„ „

Considering that pure oil of turpentine has at 15° a specific gravity from 0.860 to 0.877 and that it passes over on distillation almost completely between 155 and 170°, the figures mentioned above give a fairly accurate idea of the extent of the adulteration. This is especially evident from the boiling characteristics, as only 37.6 per cent. passed over at the above-mentioned range of temperature.

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[1]) Comp. Gildemeister und Hoffmann, *Die ätherischen Öle*, 2nd ed., vol. III, 173. — [2]) *Chem. Weekblad* **13** (1916), 692; *Chem. Zentralbl.* **1916**, II. 588.

The adulterant consisted of a petroleum fraction, which was proved among other things by the fact that when it was shaken with the same volume of cinnamaldehyde more than half the oil became separated. Cinnamaldehyde does not mix with petroleum, whereas oil of turpentine dissolves in cinnamaldehyde to any extent.

That really good oil of turpentine is still obtainable was amply proved by the quality of the commodity forwarded after raising objections.

Camphor oil is often used as a substitute for oil of turpentine. E. Coen[1]) recommends the following test for detecting it in oil of turpentine, a method depending on the presence of safrol in camphor oil. 100 cc. of the oil in question are distilled and the 5 last cc. of the distillate are mixed drop by drop with 5 cc. of concentrated sulphuric acid, whereby the mixture is to be cooled each time after adding the acid. After adding 20 cc. of water, the mixture is shaken with 10 cc. of amyl alcohol and then 5 cc. of a 20 per cent. carbonate of potash solution added. The presence of safrol is then said to be apparent by a green or blue colouring which turns to red on adding sulphuric acid. On retesting Coen's method we were quite unable to detect the colouring mentioned by him. Mixtures of oil of turpentine with safrol as well as camphor oils containing the latter only produced a dirty violet colour which turned to red on adding sulphuric acid.

Hence we will refrain from criticism until we have been able to study his original paper.

The question of obtaining turpentine products in Germany is till a matter of vital importance, hence the litterature on this object has become exceedingly voluminous and several long articles about it have appeared which are suitable for our present *Report*.

E. R. Besemfelder[2]), in an article entitled "The Sources of Resin in Germany", attempts to answer the question whether we possess sufficient sources of resin in Germany, if these are utilized in an efficient manner, and finally, if we are able to become independent of foreign countries which have hitherto supplied us.

Rosin and oil of turpentine, both substances which are almost indispensable in many arts, such as the varnish, paint, soap, munition, and paper manufacture, are solely produced by pines and firs, in spite of all attempts to find substitutes by distilling coal. According to the survey of woods and forests in the year 1900, the total area covered by them in Germany amounted to 13 995 869 hectares[3]), of which 9 451 069 hectares were covered by pines and firs. Of the high forests, 2 571 952 hectares consisted of hard wood and 8 407 267 hectares of conifers, and of the latter 5.6 million hectares were pines. The total area covered by high forests amounted to 10 979 219 hectares, the remainder consisting of copses, brushwood, and plantations.

According to an estimate made in 1913, 12 462 000 tons of pine and fir wood were available in Germany. The question is now as to how the turpentine products contained in this enormous quantity of wood, can best be utilized. Besemfelder proposes two solutions for this purpose, one being the quick-ripening of the wood, the other the extraction of all wood waste from root-stocks to sawdust.

According to the quick-maturing process which the firm of Schilde, of Hersfeld, Hesse-Nassau, have worked out, wood taken fresh from the forests and freshly sawn, is turned into quite mature wood, capable of being worked in every manner, in the course of 3 days. The resin, produced from conifers according to this method, remains

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[1]) *Annali Lab. chim. centr. della Gabella* **7** (1914), 99; *Journ. de Pharm. et Chim.* VII. **12** (1915), 395. — [2]) *Chem. Ztg.* **40** (1916), 997. — *Chem. Industrie* **40** (1917), 11. — [3]) 1 Hectare = 2 1/2 acres.

easily soluble, whereas if exposed to the air for only 26 hours, it looses so much in solubility that 40 per cent. less than in a fresh state are absorbed by the solvent. The freshly hewn wood, immediately on leaving the saw, is introduced into a boiler, capable of being closed and heated, and locked therein in a steam-tight manner. An evaporator, fixed close to this dry boiler, now supplies an uninterrupted stream of an auxiliary vapour, preferably with a constant boiling point, and from an organic liquid not miscible with water, and in the case of coniferous wood being a good solvent for resin and oil; at the same time the dry boiler is heated continually in order to prevent the condensation of the auxiliary vapour in the pores of the wood and a too extensive extraction of the latter which would affect its strength and resistance. As soon as the vapour has begun to act only a short time, the sap of the wood begins to flow and indeed it streams from the lower discharge pipe of the boiler in a liquid state as well as in the form of a mixture of steam and of auxiliary vapour rising from a channel at the top of the dry boiler and then into a condenser where the mixture is condensed. The liquids then are generally run into an automatic separating vessel, in which the water, charged with the exuded and volatile components of the sap, separates, when at rest, completely from the water-repelling auxiliary liquid, according to the specific gravity, so that the water flows in an uninterrupted stream from the one discharge pipe of the separating vessel into a reservoir, whereas, on the other hand, the condensed auxiliary liquid, charged with resin, oil of turpentine, fat, &c., runs out of the 2nd discharge pipe of the separating vessel and then back again to the evaporator. At the end of the drying process the resin, &c., which has been detached from and dissolved out of the wood remains in the evaporatoy as an "extract" after the auxiliary vapour has been distilled off. This "extract" is then run off and preferably transferred to plant erected in common for working up such residues. Investigations as to the value of the sap-water have still to be made. Theoretically, a small quantity of solvent would suffice to render an unlimited quantity of wood ripe for manufacture, but in practice one must reckon with a loss of solvent of 1 per cent. of the rough wood weight. The distilling process is continued until no more water runs from the separating vessel, which in some cases dribbles slightly. Besides this criterion for the termination of the drying process, there is another one, namely that the temperature in the drying boiler only reaches the boiling point of the auxiliary liquid when all the moisture has been driven out of the wood. As long as the latter still contains much moisture, the temperature of the vapour mixture remains fairly constant at a certain level below the boiling point of the lowest-boiling component of the vapour mixture, as for instance at 75°, when the vapour mixture consists of the vapour of trichlorethylene, boiling point 88 to 90°, mixed with steam. It then rises quite gradually, as the water gives out, and only mounts up to 88° when the last drops of water have escaped. Wood, dried in this manner, is also perfectly sterile, nor does it contain any living insects or their larvæ or eggs.

On an average there might be obtained about 1 per cent. of rosin, oil of turpentine, fat, &c., accordingly from 12462000 tons of resiniferous wood, produced in Germany, about 124620 tons of extract which would about cover the German demand for rosin and oil of turpentine. If the value of extract is taken at ℳ 1.— per kilogramme. Germany could obtain, solely by adopting this rational quick-ripening process for wood, a value amounting to ℳ 124620000 from the extract. Besides which, no consideration has been paid to the value possibly obtainable from utilizing the sap-water.

Germany, however, has other sources of resin at her disposal besides the method just described, as Russia, Austria, and Hungary together supply her with 4622570 tons

of wood a year for building and furniture-making purposes. A continuation of the exports from Austria and Hungary may be depended upon and it is probable that the rich woods in the occupied zone of Russia will continue to supply us. This would mean an additional production available in Germany of 46225,7 tons of extract valueing 46225700 ℳ.

Even if this new process were only introduced gradually, we have another supply of resin and turpentine products, *i. e.,* all the coniferous wood imported for the production of mechanical wood-pulp and cellulose, to the amount of 1217993 tons a year, besides all roots and brushwood, without counting the chips, sawdust, and waste from coniferous wood. The latter, owing to their being produced in a disintegrated state, could all be turned over to an extracting process, and in point of fact to a much more exhaustive one than the quick-maturing wood process, as the latter is particularly adapted not to exhaust the wood, whereas the above-mentioned wood-waste and, in fact, a great part of the home coniferous wood production (for instance that used in the production of paper) actually demands a complete extraction of resin and fat.

Of the supply of root- and brushwood, about 2,319 million tons could be used for extraction which would give approximately 69570 tons of extract. On the whole about 240415,7 tons of extract could be depended upon.

It may be calculated that only about half of the extract obtained consists of rosin (and oil of turpentine) and the remainder of fat. Our production of rosin and oil of turpentine amounts accordingly to about 120200 tons, which cover the peace demand of Germany hitherto completely according to Wislicenus and Keutgen. The fat consists principally of oleic acid. The oil yield from coniferous trees corresponds to almost a quarter of the oil and fat quantity imported with oil fruits.

Sawdust itself, by being freed from rosin, &c., develops a number of qualities which make it more suitable for technical purposes. Under these conditions it is perfectly dry and is a much better absorbent than before, which makes it much more valuable for packing purposes, filling dry batteries, &c. It can also be used as filling material in the production of fodder without any detrimental effects owing to presence of rosin.

The author gives no information as to the yield and quality of the oil of turpentine. However, it must borne in mind that oil of turpentine is a pathological product. Whether the oil, produced according to the above-mentioned process, contains pinene and what qualities it has, does not appear from the abstract. Obviously it must be contained therein if it is to be employed in the manufacture of artificial camphor, as the author expects.

In connection with this article, G. Austerweil[1]) refers to the new sources of rosin which were opened up during the last year in the Austro-Hungarian Monarchy, in the adjoining countries, and in the occupied territory. These consist of: —

1. The exploitation of the resin-supplies produced by the woods of the occupied territories. 2. The utilization of such resin-supplies for the production of colophony which were hitherto not available for that purpose. 3. The manufacture of colophony by means of the extraction of roots, such as are left in the ground by a rational management of forests.

Outside Austria-Hungary about 1000000 trees were tapped, nearly all belonging to the white-fir species *(Pinus silvestris),* which in comparison to the black-fir *(Pinus austriaca Laricio)* have a much lesser resin output, so that the yield was not very high.

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[1]) *Chem. Ztg.* **41** (1917), 233.

As well as in Germany, the resin scrape has been collected in Austria. Although it is mentioned in litterature that pine resin is not suitable for the production of colophony, a most simple process for the production of colophony from pine resin has been successfully worked out. The resin is extracted by hot benzene, the solution is then treated accordingly and filtered, whereupon the benzene is distilled off in a column-apparatus and finally the oil of turpentine[1]) is driven off from the colophony by the aid of steam and a vacuum. The average yield of colophony from such resin scrape amounts to 55 to 60 per cent., besides 2 to 3½ per cent. of oil of turpentine. This kind of resin is not produced by tapping or chipping the trees, but by the action of the deer, and only necessitates collecting.

A rational combination of the root extraction industry with the normal exploitation of forests was introduced in Hungary for the first time last year in such a manner that the roots left behind in the yearly wood clearance scheme which are useless for wood culture were turned over to the wholesale manufacture of colophony and of oil of turpentine. Erecting a root extraction plant for the purpose of producing colophony and oil of turpentine every year from the output of roots on a commercial scale, has only succeeded economically at such places where every day at least ¾ to one carload is at disposal. The stockwood is worked out by means of explosives, preferably with a chlorate explosive. Benzene is used as a solvent for extraction, occasionally also trichlorethylene. The disintegrated wood is freed from oil of turpentine by means of steam alone or with help of a vacuum, before being extracted. The yield of oil of turpentine amounts to 1 per cent. with white fir and about 2 p. c. with black. The wood-waste, freed from oil of turpentine, produces as an extract a viscous resin which consists of about 90 per cent. colophony and 10 per cent. of a product resembling oil of turpentine, in which, however, the terpene alcohols predominate. The extract is introduced in a liquid molten state into a vacuum distillation apparatus in which these 10 per cent. volatile components are driven off, this being the most difficult part of the whole process. The volatile compounds are united with the oil produced by steam from the wood-waste, and appear on the market as wood turpentine oil. This product finds favour in various branches of industry. Owing to its high terpineol content it is superior to American oil of turpentine for use as solvent in the varnish industry.

Three such establishments are in operation in the Austro-Hungarian Empire. The products they put on the market resemble ordinary oil of turpentine and colophony, but differ from them in some respects. The extraction of the roots is conducted according to two different methods: 1. either the wood is first disintegrated and the volatile oil of turpentine driven out of it before being treated with solvents, the extraction taking place afterwards, or 2. the wood is extracted with solvents directly. If the first method is adopted, a yield of ¾ to 1½ per cent. with white fir wood and 1¼ to 2½ per cent. with black fir of normal oil of turpentine, slightly yellow in colour, and having the specific gravity of 0.876 to 0.879 at 15° is obtained which, apart from a slight musty odour, owing to traces of fenchyl alcohol, can be, according to Austerweil, regarded as fully good oil of turpentine. One single rectification is sufficient to produce therefrom a first-class water-white product of the specific gravity of 0.861 to 0.867 which, besides containing pinene, camphene, and dipentene, also shows a small amount of terpineol and fenchyl alcohol. The only manner it differs from American and Austrian oil of turpentine (from *Pinus Laricio*) is that it contains more high-boiling constituents then these, *i. e.*, that the portion which passes over above 160° is rather greater than

[1]) Probably only a product resembling oil of turpentine, like wood oil of turpentine.

with ordinary oil of turpentine. A similar product is current on the market as "Wood spirits of turpentine". According to Austerweil, the German term *Holzterpentinöl*, adopted by Gildemeister and Hoffmann in their text-book (2nd German edition, vol. II, p. 100) would correspond exactly to the above-mentioned English expression[1]).

It is very difficult to obtain a really brittle colophony from roots, as besides ordinary volatile terpenes great quantities of less volatile terpene alcohols are present in it, without counting the oxidized terpenes which are produced from the existing normal terpenes during the process of disintegrating the wood for extraction, owing to the great surface it offers to the oxygen of the air and to the resulting rise of temperature.

These high-boiling products which are alone not easily volatilized are retained by the abietic acid with much tenacity and can only be driven out after prolonged distillation. At 164°, under a pressure of about 90 mm., products were formed from the crude resin which boiled between 190 and 230° and consisted, along with small quantities of cineole, pinole, and fenchyl alcohol, mainly of terpineol which could be identified by its nitrosochloride. The oil thus obtained, of the specific gravity of 0.934 to 0.937, consists mainly of terpineol and corresponds to the substance known in America in trade as "Pine oil".

C. G. Schwalbe[2]) adopts in the main a very sceptical attitude towards Besemfelder's above-mentioned observations. He calculates that with a yearly consumption of 20 million cubic meters of coniferous wood 560000 cbm. of apparatus space are necessary; of the size, mentioned by Besemfelder, at least 8000 installation plants for the quick-maturing wood process would be required which could hardly be obtained in the course of the next years. He is also very sceptical whether the resin could be extracted fairly completely from wood for building purposes and adds that the experience gained in America with the extraction of resin from disintegrated wood is distinctly against this assumption, which proves that, if the pieces of chopped wood exceed certain dimensions, the extraction is no longer complete.

Besides this, to deprive Scotch firs of their resin is not exactly an advantage for many purposes, as the good qualities of this wood for the manufacture of casks and vats are mainly dependent on its richness in resin.

He also finds that a too high estimate has been made of the figures for the yield in resin as well as of the value of the extract.

There are other facts also to which Besemfelder, in Schwalbe's opinion, has not paid any attention, for instance the extraordinary bulkiness of the material, which not only would cause an enormous increase in the cost of transport, but would also prevent cheap extraction. In this respect experience in the U. S. is instructive as a warning from exaggerated hopes, where the extraction of wood rich in resin only begins to be a paying concern when it contains at least 15 to 25 per cent. of it. Such rich material only occurs very rarely in Germany.

With due consideration to the whole matter Schwalbe appears to think it improbable that Besemfelder's far-reaching wishes and plans concerning the production of rosin, turpentine, and fat are likely to be realized by means of the extraction of waste wood. He recommends on his part, as a much cheaper method of getting at the resin content of such refuse, the exploitation of wood really rich in resin, such as the roots

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[1]) In the work mentioned, however, the opinion is expressed that "wood turpentine oil" can by no means in all cases be regarded as a substitute for gum spirits of turpentine owing to the presence of high-boiling constituents observed also in the Austrian product. — [2]) *Chem. Ztg.* **41** (1917), 257.

of Scotch firs, and in point of fact suggests that this procedure is to be combined with the manufacture of cellulose from wood[1]). If such wood is boiled with caustic soda under pressure, turpentine might be blown off with steam towards the end of the process and in the liquor the rosin dissolved will be found, besides obtaining the cellulose of the wood[2]).

Besemfelder[3]) rejoins that he made no pretensions to free the whole supply of coniferous wood from resin, he laid special stress on the fact that the quantity of extract produced, rose and fell with the amount of the wood surface introduced into the drying apparatus.

Besides this, according to Schwalbe's calculations, not 8000 but 4000 boilers would be necessary for the rapid-maturing wood process.

Besemfelder thinks, however, that Schwalbe's assertions prove that by utilizing the various sources of resin, especially by tapping our coniferous trees, it will be possible to cover the German demand for turpentine products.

For obtaining turpentine by tapping, H. Wislicenus'[4]) process, which we already reviewed in our last October *Report*, seems to be the most practical. To judge from an article[5]) of his on turpentine production from Scotch firs by means of open and of closed tree lesions, others seem to prefer the old incision method.

As Wislicenus remarks, it is of vital importance in Germany's present condition to obtain the gross demand for turpentine oil and good resin from her own forests. Whereas on the one hand it was possible to obtain easily sufficient less valuable gutter and scrape resin, very poor in oil of turpentine, according to the cheap and simple blazing method, as practiced in the Saxon *Vogtland* from time immemorial, which is now being utilized again, the problem had hitherto not been solved of producing good oil of turpentine in sufficient quantities as well as colophony of a quality such as the resin-working and nearly always resin-distilling industry demands. The value of the crude resin diminishes considerably according to the length of time the crusts of resin are formed on the trees owing to the oxidizing effect of the air. The so-called "wild resin", being mostly old and brittle, which had to be collected in 1915 from the abrasions, produced by deer on pines, was positively refused by the resin-working industry owing to its having become unsuitable for distillation purposes from the effect of the air and from admixture with impurities of all kinds. When open incisions are made, the value of the resin diminishes very quickly; but where the bottle system is employed, a product of very great value is obtained. A sample of turpentine of this kind distinguished itself on examination in our laboratory by its very light colour and the total absence of mechanical impurities most favourably from the ordinary commercial products. On being distilled with steam, 32 per cent. of oil of turpentine were obtained. After remelting the residue, a light yellow colophony was produced whose most promising quality is apparently its light colour and which accordingly would be more suitable for some purposes than the ordinary more or less brown commercial product. The physical and chemical qualities of the oil proved it to consist of α-pinene in the main. It is characterized by a mild, pleasant smell and can compete in every way with the very best trade samples.

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[1]) Comp. Schwalbes article *Harz und Terpentin aus deutschem Walde* (*Zeitschr. f. Forst- und Jagdwesen* 48 [1916], 99). In this article the author expresses the opinion that at present it cannot be foreseen if and to what extent a German rosin and turpentine industry, dependent on the resin-tapping, &c., of the living Scotch pine, can the founded with paying results in times of peace. — [2]) In this article the terms turpentine and oil of turpentine are confounded! Comp. p. 78, foot-note 1. — [3]) *Chem. Ztg.* **41** (1917), 258. — [4]) *Chem. Ztg.* **40** (1916), 559; *Report* October **1916**, 53. — [5]) *Forstliche Wochenschrift Silva* **1917**, N° 11.

The incision-method of obtaining resin and turpentine from Scotch pines demands in its modern form difficult drilling work with 5 cm. drills and in point of fact it is not sufficient to bore one hole for each outlet, as the experience gained in the Government-General of Poland in 1916 proved. The small and only a few cm. deep bore-holes for the bottle-turpentine-collection with the help of a 3.4 cm. drill demand less individual work, even if one adds to them Kubelkas' side channels with a 12 or 15 mm. drill, whereby it is quite useless to go any deeper.

Wislicenus discusses besides the pros and cons of both methods of producing turpentine and finally publishes a memorandum card as a guide for obtaining turpentine from Scotch pines with guarded incisions on which the various manipulations are illustrated with the aid of photographic reproductions of the tools employed[1]).

Some of the opposition that Wislicenus' method met with reminds of the difficulties the introduction of the "cup-and-gutter" method encountered and about which H. Herty[2]) reports. Owing to the references made by the well-known technologist O. N. Witt on the great damage the Naval Stores industry was suffering from the turpentine production methods generally employed, Herty approached this subject first of all by correspondence and then paid a visit to a turpentine farm in Southern Georgia, where he found that the matter was much more serious than was generally known in the U. S. The result of his investigations was the working out of the "cup-and-gutter" system[3]). The U. S. Bureau of Forestry supported him vigorously and decided to enable Herty to make experiments on a big scale. These were conducted early in 1902 on a turpentine farm in South-eastern Georgia belonging to the firm of Powell, Bullard & Co. The latter put the best trees at his disposal, whereas the Bureau of Forestry offered the necessary implements and apparatus. The trial enterprise was executed just in the same manner as the regular routine prescribed. The experiments only applied to the "box"-question and to the possibility of substituting the boxes by cups, whereas the other methods of production were to be retained. Four series of tests were made simultaneously for the sake of comparison, and in point of fact one series on virgin pine stock, the other three according to the ordinary process on tracts of forest which were worked for periods varying from one to three years. By this means comparative data were available as to the influence of the "box" and "cup-and-gutter" system respectively on the trees and on the production of turpentine and profit of the owner of the forest respectively.

So far matters went smoothly, but then difficulties cropped up, especially in reference to the workmen. The niggers who worked in the turpentine woods came forward with strangely conservative demands, declaring that what they termed derisively "the flower-pot" system was more an occupation for women and children, but quite beneath the dignity of full-grown men! Finally they were persuaded to give the new system a trial and they gradually got used to it and learnt to appreciate its advantages. The principle interest centred in the experiments with virgin Scotch pines with which both methods could be applied under quite equal conditions. Contrary to expectations the first yield of the cups (in April) showed a reduction, but the second yield in May and all the following ones proved beyond dispute how advantageous the new system was in comparison to the old one.

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[1]) Comp. the same author's paper, *On the production of German Turpentine by means of closed tree-lesions*, in *Naturwiss. Zeitschr. f. Forst- u. Landwirtschaft.* — [2]) *Journ. of the Franklin Institute* **1916**; *"Der Mörser", Organ des Schweizerischen Drogisten-Vereins* **16** (1916), 553, 565, 577, 605. — [3]) See Gildemeister and Hoffmann, *The Volatile Oils*, 2nd ed., vol. II, p. 60.

The distillation of the turpentine from the following yearly yields according to the cup-system produced a fine, light-coloured rosin as a residue which distinguished itself most advantageously from that produced by the box-method. Besides this the cup-system showed itself to be admirably adapted for obtaining turpentine from trees with higher-reaching chippings.

The results of the first experimental year were made known to the owners of the forests and aroused much interest, but this soon diminished and could only be kept alive by further efforts and by drawing attention to the commercial advantages of the cup-system. Finally it was decided to give the cup-system a trial to a certain extent. The good results obtained in the second year brought about a notable advance in the matter, the box-system was gradually relegated to the "lumber-room", the new method gained supporters and friends on all sides, so that finally the cup-method won the day over the wasteful box-system at least in principle. Nowadays the authorities in the Naval Stores regard the box-system as quite antiquated and various sorts of cups are in use and current in trade. Even the railway companies of the Southern States have gone so far as to arrange cheap freight-rates for the carriage of all the articles needed in the cup-system.

The reports of the results of the practical application of the cup-system in coniferous woods gave proof of much larger yields in turpentine everywhere, but also pointed out as a drawback that more extensive "chipping" was needed to fill the cups at the beginning of the year than was necessary according to the old box-method. This was a confirmation of the unexpected lesser yield in the trials with the cups at the end of the first month. The underlying cause is probably the following: —

According to Tschirch's opinion, the resin-ducts of every normally developed pine tree contain a resin which is formed in the living tree during its process of growth and is accordingly a purely physiological product. Tschirch calls these ducts "primary resin-ducts". They only produce very little crude turpentine on being "chipped". Immediately after the chipping, however, numerous resin-channels are formed in the fresh outside wood round the injured part and an "Anastomosis" (ramification) of the vessels begins, from which crude turpentine flows abundantly as a balm for the wound inflicted. This exudation is accordingly a pathological product and Tschirch calls the channels which produce this gum "secondary resin-ducts". They require for their full development about 5 weeks. This theory of Tschirch also explains satisfactorily why the cup-system gave a lesser yield in the first test-month. With the box-system, after the "boxes" had been cut out round the trees some time elapsed before the chipping operation was performed. In the meantime, numerous secondary resin channels were formed which gave off liquid resin abundantly on being chipped. With the cup-system, on the contrary, chipping was begun with directly after the first incision and as a result secondary resin channels were not formed first of all, accordingly the exudation was scanty, but rose, however, very much after some weeks, when these resin channels had appeared round the incisions and then discharged liquid resin abundantly. Accordingly, it is imperative in the cup-system after fixing the pipes to the cross-cuts of the trunks, which are intended to conduct the liquid resin into the cups, to begin chipping immediately, then to leave the trees alone, and only to chip again after a period of 5 or 6 weeks. By this means a full development of the secondary resin-ducts round the incision and an abundant flow of turpentine is warranted. Practical experience proved Tschirch's theory to be correct, and the cup-method produced under these conditions much larger yields, so that theory had effectively supported practical experience.

Experiments have further given proof of the fact that a careful treatment of the trees, especially care in chipping, has decided advantages and leads to a better yield of turpentine.

One disadvantage of the cup-method is caused thereby that frequently very thin trees are worked, which was not the case in the box-system. The following considerations are a warning against this practice: 1. Possible damage to, or destruction of, the trees. 2. Such processes being unremunerative.

It has been definitely proposed to substitute the distillation on a small scale of the individual turpentine-farms by a distillation on a large scale at a suitable place, where the crude turpentine can be brought to in a tank-waggon. The future must indicate if such a proposal is feasible in practice.

Further particulars about the qualities of the turpentine or oil of turpentine contained in coniferous wood are to be found in a publication by C. G. Schwalbe[1]) on the resin of pine trees and especially of Scotch pines. The voluminous literature on the resin content of coniferous trees treats principally of the so-called "pathological" resin which exudes or is formed when a tree is injured. Only a small part of these investigations refers to the normal resin of the living tree or to the resin contained in wood that has been felled.

In Schwalbe's paper experiments are mentioned in detail which had the object, by a close investigation of the resin in fresh and mature wood, of obtaining information as to its qualities and composition.

The attempt to determine the quantity of turpentine obtained in the alcohol and ether-extracts lead to a most surprising result. One is accustomed to regard the crude resin of coniferous trees as a mixture of turpentine oil and of special resinic acids, especially with regard to those kinds of resin which exude from the trees on being injured. This notion concerning the turpentine yield of crude resins has also been transferred to the resin in the undamaged wood substance of coniferous trees.

In order now to determine the real yield of turpentine[2]) in resin-fat mixtures in the first instance from mature wood, an attempt was made to drive off the oil of turpentine by the aid of steam. Contrary to all expectations, no trace of oil of turpentine could be found in the distillate as a separate oil layer. As possibly other volatile components of the crude resin might have facilitated the solubility of the oil of turpentine in water, the distillate was extracted with ether, the latter evaporated, and the remainder concentrated at a moderate temperature, but even then no residue was obtainable!

This result was all the more surprising as it is notorious that quite considerable quantities of oil of turpentine are obtainable as a by-product of the soda-cellulose manufacture from wood that has been stored for several years. Possibly the presence of turpentine or turpentine oil might be caused in this case by the portion of the wood which chanced to be examined having lost its (oil of?) turpentine content during the protracted storage period, had it not been for the fact that the examination of the resin-fat extract from quite freshly felled wood lead to exactly the same result, namely total absence of (oil of?) turpentine. If, however, such freshly felled wood was subjected to a treatment with caustic soda solution under pressure and heating up to 170°, oil of turpentine was easily detected in the current of steam that was blown off.

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[1]) *Zeitschr. f. Forst- u. Jagdwesen* **47** (1915), 92. — [2]) In this paper the terms turpentine and oil of turpentine are perpetually mixed up. Unfortunately, the text does not always allow one to form an opinion if the oil or the gum is referred to. Comp. page 78, footnote 1.

The quantity of (oil of?) turpentine from quite freshly felled wood, found in ordinary pines under these circumstances, amounted to 0.11 per cent., in the case of Scotch pines, 0.8 per cent.

Proof was further obtained that oil of turpentine was present in the blown-off steam when the wood which had been extracted with ether was again heated with caustic soda solution under pressure. Hence one is forced to the conclusion that oil of turpentine is practically preformed in freshly felled wood only to a very small extent, in matured wood not at all really, but that on the contrary it is only created as a decomposition-product from wood when the latter is subjected to as high a temperature as 170° and when caustic soda solution is employed as an agent of decomposition.

The fact also that in the cellulose industry oil of turpentine is only given off when the heating vessels have reached the maximum pressure of 6—8 atmospheres and simultaneously the maximum temperature of 170°, quite corresponds with these observations, whereas oil of turpentine ought to pass over with steam at considerably lower temperatures, if it were really present as such in the wood.

It is well known that the mother plant of Indian oil of turpentine is *Pinus longifolia*, Roxb. which is found in British India. The Imperial Forest Department of India has published a voluminous report about this pine by R. S. Troup[1]) which has also been the subject of discussion in various scientific papers. *Pinus longifolia* grows in the Himalayas on the Siwalik hills from Bhutan to Afghanistan; in the Eastern districts in heights reaching 6000 feet, but occurring rarely there above 5000 feet; in the West, however, it is to be found at an altitude of 7500 feet. It has frequently been cultivated in the plains also, outside its natural habitat, even at Calcutta. Besides it has also been transplanted to the Nilghiris, on the plateau of Raigarh, in the Balaghat district, and elsewhere.

According to the most recent calculations, the area covered by *Pinus longifolia* amounts to about 3370 sq. miles. Besides, however, there are also woods of it in Sikkim, Bhutan, Nepal, Mandi, in the border states, and in Afghanistan, the extent of which is as yet unknown.

Pinus longifolia is gregarious, which facilitates the collection of the turpentine. In some districts it is often found together with deciduous trees. It adapts itself to the most various soils, is more susceptible towards low temperatures than towards high ones and does not thrive accordingly in too elevated districts, but on the contrary in the hot climate of the plains.

Under favourable conditions this tree attains a height of 180 feet, only reaching 20 feet, however, under unfavourable conditions.

Nothing is yet known about the oil produced by the needles; attempts to utilize them for the production of oil do not appear to have been made hitherto.

Occasionally the tree throws off its needles, especially in dry, hot districts, as for instance in the hilly region near Kangra and below Naini-Tal, generally, however, it retains them for about 17 months, exceptionally even for 2 or 3 years. At unsuitable spots the branches cast off their needles in the beginning of March, generally, however, not before April or May. By the end of June or July the last have dropped off; the new ones, however, soon grow in their place and are 3 to 5 cm. long when old ones are cast off, attaining their full size in August or September.

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[1]) *Perfum. Record* 7 (1916), 243, 280.

Every 2 or 3 years a good seed-year occurs which is of importance for the propagation of the species. Under normal conditions, *Pinus longifolia* does not bear any fir cones before reaching its 30th year; trees are by no means a rarity which have attained even their 40th year without bearing fruit. Trees planted on an experimental field at Dehra Dun only began to bear fruit at 30 years of age. On an average, the trees reached a height of 79 feet with a circumference of 3 feet 3 inches. Germination tests proved that 81 to 93 per cent. of the seeds were capable of germination. For old, over-ripe trees this capacity only amounted to 35 to 60 per cent. 284 cones gave an average seed-content of 51 seeds; the real number varied from 31 to 105 seeds per cone. The seedlings attain a height of 6 feet at 3 years of age, after 10 years they are about 5 feet high.

Pinus longifolia demands a great deal of light. In spite of this, however, the seedlings require a certain amount of shade, especially when they are quite young.

Under the condition that the forests are protected from conflagrations and from grazing animals, their continued existance in the case of their being utilized for industrial purposes depends on the possibility of their propagation by seeds. In some districts, however, the mortality amongst the seedlings is very great indeed, especially on southern slopes.

Porcupines and certain species of birds also cause much damage. Amongst beetles, the wood-borers, *Ips longifolia, Polygraphus longifolia, Platypus biformis* and the weevil *Cryptorrhynchus Brandisi* are particularly dangerous. Besides these, a creeper, *Rosa moschata,* is harmful. The fungi, *Peridermium complanatum, Hysterium Pinastri,* and *Tramentes Pini,* frequently attack the tree; and *Loranthus longiflorus* is a regular parasite of this kind.

The tapping is performed according to the methods in use in France.

The Italian stone-pine, *Pinus Pinea,* L.[1]) is to be found along all the coasts of the Mediterranean and on neighbouring hills, in North Africa, on Madeira and the Canary Isles, in Portugal, in Spain, up to a height of about 1000 m., further in Western Italy, forming there, for instance, the celebrated pine wood near Ravenna; in Illyria, Dalmatia, the Peloponnesus, in Crete, in Bithynia, Macedonia, and Asia Minor; it is further much cultivated[2]) in other countries having a climate resembling that of those mentioned above. As turpentine is obtained in Tuscany as well from *Pinus Pinaster,* Sol. (black pine) as from *Pinus Pinea,* without, however, distilling both kinds separately, and as both oils have very different properties, F. C. Palazzo[3]) produced from turpentine of *Pinus Pinea* which had been collected under his own personal supervision the volatile oil by the help of distillation with steam under a pressure of $2^1/_2$ to 3 atmospheres. The constants of the oil were the following: $d_{15°}$ 0.8473 to 0.8507; α_D — 93.80 to 97.30°; $n_{D20°}$ 1.4731 to 1.4737; soluble in about 5 vols. of 90 per cent. alcohol; 85 to 90 per cent. of the oil boiled between 174 and 180° and consisted of laevo-limonene (boiling point 175 to 176°; $d_{20°}$ 0.8432; $\alpha_{D20°}$ — 120.36°; $n_{D20°}$ 1.4720).

The production of turpentine in Tuscany is not of great importance. The production of the oil described above is recommended because, owing to its limonene content which is utilizable for various technical purposes, it is said to be more valuable than ordinary oil of turpentine.

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[1]) This pine (*Pinus Pinea,* L.) is mistaken in some books and by the general public for the sea-pine (*Pinus Pinaster,* Sol.); the same seems to be the case in Italy, which is probably the reason why both kinds are distilled together. — [2]) L. Beissner, *Handbuch der Nadelholzkunde,* 2. ed., Berlin 1909, p. 423. — [3]) *Annali Chim. appl.* **6** (1916), 135; *Journ. Soc. chem. Industry* **35** (1916), 1122.

Wood turpentine oil and similar products. Cellulose is produced generally for the manufacture of paper according to two different processes, *viz.*, the sulphite and the soda process. In Germany, the former process is principally worked, although the waste liquors which are produced thereby, for which hitherto a practical application has not been found, are a cause of great annoyance to the manufacturers. The soda process has the advantage of permitting the utilization of wood rich in resin (from coniferous trees); the manufacturing costs, however, are higher and it is by no means easy to obtain a licence for manufacturing, as the smell of the cooker liquors is very disagreeable. The losses of soda are covered in this process by the addition of sodium sulphate, after which extraction takes place in the usual manner and burnt lime is used as a caustic. The sodium sulphate is hereby partly transformed into sodium sulphide which acts on the fibres in the same manner as caustic soda solution; accordingly the method is also called the sulphate process and the cellulose produced thereby "sulphate cellulose"[1]). A by-product resembling oil of turpentine is obtained according to this method, which originates from the portions of the wood rich in resin. This oil is blown over by the steam after the termination of the boiling process and is then condensed in a suitable receptacle outside the cooker. According to O. M. Halse and H. Dedichen[2]), the quantity of oil produced in this manner varies to a great extent, as it depends on the content of resin compounds in the wood. Ordinary pine wood generally gives a yield of 1 to 1.5 kg. per ton of cellulose, whereas wood from Scotch pines produces as much as 10 kg. per ton of cellulose.

In a crude state the oil has a detestable smell which is caused by the presence of mercaptanes and of other organic sulphur compounds, *e. g.*, of methyl sulphide. For using it as a substitute for ordinary oils of turpentine, these substances must be removed as much as possible. A number of patent processes are already known for this purpose which mostly depend on a careful oxidation of the sulphur compounds, for instance by the effect of air or light. In some works also fractionated distillation is employed, and by this means the sulphur compounds, owing to their boiling point being lower than with the oil, can be almost completely removed.

The authors examined the crude volatile oil, just as it usually comes from the works, without its having been purified. It smelt in a most disagreeable manner of mercaptane and accordingly they began by treating it first of all with an alcoholic solution of mercuric sublimate, after which they distilled it in the ordinary manner by the aid of steam. Hereby a viscous brown liquid remained as a residue, forming about 8 per cent. of the oil, and consisting of the ordinary resin-like oxidation products of oil of turpentine.

The purified oil was colourless and clear; it smelled of fresh wood, but was otherwise quite like gum oil of turpentine. The constants observed were: $d_{15^\circ}^{15^\circ}$ 0.8657 and 0.8641; α_{D20° + 17.05 and + 18.55°; n_{D20° 1.47151 and 1.47270. The oil dissolves in 5.5 vols. of 90 per cent., 25 vols. of 80 per cent., and 70 vols. of 70 per cent. alcohol. It boiled mainly between 154 and 155° and between 167 and 168° and consisted principally of *d*-α-pinene, which was recognised as such by oxidation to pinonic acid. The semicarbazone had its melting point at 203°. The presence of β-pinene was effected by the formation of nopinic acid, melting point 123°; it is present in this oil in a *dextro*rotatory form, whereas hitherto it was only known as a laevorotatory product. The authors are unable as yet to determine whether the β-pinene exists in a dextro-

[1]) O. Dammer, *Chemische Technologie der Neuzeit*, Stuttgart 1911, vol. III, p. 8. — [2]) *Berl. Berichte* 50 (1917), 623.

rotatory form already in the wood or whether it is the result of the sulphate-cellulose process, acting on *l*-β-pinene. Probably, dipentene also exists in small quantities in this oil.

It is curious that α-pinene which otherwise does not belong to the most stable terpenes stands the long boiling process of the cookers in the sulphate-method so well, whereas in the sulphite-cellulose process it is converted into the more permanent hydrocarbon *p*-cymene. In this process the wood is boiled with acid sulphite liquor and in point of fact the sulphurous acid is generally added in the form of a calcium or magnesium bisulphite solution. Z. Kertész[1]) describes the formation of the volatile oil in the manufacture of sulphite cellulose as follows: — After the termination of the cellulose boiling the still unused sulphurous acid gases liberated are passed over into a so-called mixing reservoir which contains fresh tower-liquor whose sulphurous acid content is increased by the absorption of the sulphurous acid gases. Besides sulphurous acid, much water vapour, acetic aldehyde, acetone, methylalcohol, furfurol, volatile oils, formic and acetic acids are condensed. The fresh tower-liquor is diluted with injection-water, the other condensation-products mix with the tower-liquor, on whose surface the volatile oils accumulate and thereby prevent the oxidation and loss of sulphurous acid. The liquor which has been carried over by the gases, the so-called overwash-liquor, also accumulates in the tower-liquor. Part of them, especially the volatile organic compounds, which are generated in the cookers in considerable quantities at an elevated temperature, stream over into the fresh cooking-liquor and circulate in this manner from one cooker to another. The compounds just mentioned are, however, only represented by tolerably equal quantities, because part of them are lost in the waste liquor and during the blowing-off of the cooker.

The volatile oils formed in the sulphite process originate from the resin of the pine wood. Schwalbe and Sieber[2]) have ascertained that from pine wood which has been stored for some time one can obtain by means of an ether-alcohol mixture 1 per cent. of substance, from fresh wood, however, 1.6 per cent. According to Sieber's[3]) latest publications, the pine wood contains 0.6 per cent. of resin or ether extract. 4.2 per cent. of this collects, owing to the boiling process, in the waste liquor or in the condensation products, accordingly one ton of cellulose contains on an average 0.6 kg. of resin products which are probably generally represented in the form of a volatile oil. Bergström estimated the quantity of oils obtainable at the figure of 1 to 1.5 kg. per ton; according to Kertész' observations, the quantity produced varies considerably and in fact depends on the varying resin content of the wood and on the manner the cooking process is conducted.

The volatile oils of the sulphite-cellulose manufacture were first examined by Klason[4]) who recognised cymene as being the main product. Bergström[5]) believed in the presence of terpenes, sesquiterpene, diterpene, and of terpene alcohol, without, however, succeeding in isolating any of them. Later on, he extracted an inactive borneol, melting point 203°, by congelation from the heavy oil which, besides other compounds, contained furfurol.

The brown-coloured oil originating from the pine wood which can be collected from the surface of the cooking liquor in the mixing reservoir in a wholesale manner has also been investigated by Kertész; it still contained 0.6 to 0.8 per cent. of sulphurous acid. It can be freed from the latter and from humidity by treatment with calcium

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[1]) *Chem. Ztg.* **40** (1916), 945. — [2]) *Verein der Zellstoff- u. Papier-Chemiker, Sommerversammlung 1914*, p. 16. — [3]) *Papierfabrikant* **13** (1915), 389. — [4]) *Berl. Berichte* **33** (1900), 2343; *Report* April **1901**, 67. — [5]) *Svensk Pappers Tidning* **11** (1914), 130; *Papierfabrikant* **12** (1914), 1040.

oxide and calcium chloride. The oil has the specific gravity of 0.878 to 0.880 and boils between 150 and 310°. After prolonged standing the oil deposits a reddish-brown substance containing sulphur, which is insoluble in cymene, ether, and water, but soluble in alcohol. The combustion analysis which Kertész made of it lead to results which fairly agree with the formula $C_{30}H_{45}O_7S$. Probably this compound is formed by the polymerisation of aliphatic terpenes of less complex composition, the sulphur of course originates from the sulphur dioxide. About 80 per cent. of the crude oil consists of *p*-cymene which was identified by oxidizing it into *iso*propylbenzoic and terephthalic acids. The oil further contains a sesquiterpene boiling between 136 and 138° (9 mm.) which gives, on addition of acetic anhydride and concentrated sulphuric acid, an intense dark blue colouring and absorbs 2 atoms of bromine. Hitherto attempts to obtain a solid derivate of the sesquiterpene have not been successful. It is a light yellow, quite transparent oil, having a very slight smell, and dissolves in 4 vols. of 96 per cent. alcohol; on the whole it reminds rather of atractylene.

Besides this, the oil also contains a diterpene which turns to a dark red colour on adding acetic anhydride and concentrated sulphuric acid. It is a viscous, orange-coloured, transparent oil with a pleasant smell, from which, however, it has not yet been possible to isolate a crystalline compound.

From the crude product, cymene can be eliminated most easily by the aid of steam distillation, whereas the sesquiterpene and especially the diterpene are much more difficult to separate. One vol. of water condensed itself with 1 vol. of distilled cymene, whereas in the case of sesquiterpene it was a matter of 10 vols. and in the case of diterpene, of 100 vols. of water.

The highest-boiling portions of the oil contain a white amorphous substance, insoluble in alcohol, and melting at 67°, probably a polyterpene.

The presence of cymene in the oil can be explained by the action of the sulphurous acid on the pinene of the wood; doubtless also the diterpene and polyterpene are the products of the reaction between sulphurous acid and pinene.

The production of pine oil in the occupied Russian territory. O. Lange[1]) has published a paper bearing this title, in which he describes in an instructive manner the primitive methods for collecting pine oil in the extensive Russian forests.

One often sees on the border or in the glades of the interminable Russian forests which offer to the forester in their black pine and ordinary pine stocks a picture of incredible neglect, strange cones of earth, leaning against miserable huts, which, while far surpassing the height of the roofs, terminate in a blunt point, bust down sideways[2]). On approaching closer, one is inclined to believe one has arrived at an immense alchemistic laboratory, erected in the open air, as one sees subterranean heating apertures, glowing red-hot, and high up in the air great pipe-lines, protected in a primitive fashion, troughs which serve as coolers, in which long worm-pipes lie built in, receiving their water from the nearest rivulet. Close at hand one further observes piles of rough root-stocks and of wood, singed to a reddish-brown colour, casks, filled with black or golden-yellow liquids, skinned carcases of horses which have been transported a long distance, close to which is visible the disengaged fat of the horseflesh, neatly collected on a flat stone, finally rough sheds which serve as a protection for a few vats of liquor. All this is pervaded by a vinegar-like, tarry smell, and incredible dirt. The earth cones form the heat-protecting jackets for a large

[1]) *Deutsche Parf.-Ztg.* **2** (1916), 256. — [2]) Comp. H. Schelenz, *Zeitschr. f. angew. Chem.* **29** (1916), I. 251; *Report* October **1916**, 55.

pear-shaped cistern, built of bricks. Before charging, the upper part of this large chamber is broken out and enlarged in order to make room for the bulky black pine root-stocks which are to be distilled. The grubbed-up and sun-dried root-stocks of the black pines are chipped a bit, where-upon they are piled up in layers inside the retort which is filled up to its neck. Then the opening is built up again to its former size, a dome, attached to a long pendant pipe, is affixed, and the interstices are carefully luted with clay. The pendant tube terminates lower down in a likewise copper coil, which, as already mentioned, lies in running water. With the help of a wooden cock the distillation products are allowed to run over into the vessels standing ready for their reception. Under the accessible bottom of the walled-up cistern a woodfire, burning with a slight flame, but noted for its heat-producing qualities, is kept up for about 3 weeks which gets the necessary draught from a short, lateral chimney, but is hardly utilized to its full extent. First of all, very thin, watery, wood vinegar distils over which is simply wasted, till drops of oil become visible on the surface of the rivulet running in the ditch. Hereupon the liquid, containing more and more oil, is collected in a wooden cask through whose lower bung-hole the watery wood vinegar is occasionally allowed to run off; as soon as the cask is full, it is closed and is then fit for transport. After 8 or 12 days, when the oil, which gradually becomes darker in colour, begins to run more sparingly, the heat under the bottom of the retort is increased and after some time the distillation of the thin tar begins which primarily still contains a lot of oil from whose surface the pine oil is poured off after the liquid has stood some time; now the tar which becomes thicker and thicker but still runs off easily is conducted from the pendant pipe, eliminating the condensing tube straight into casks previously placed in readiness, and at the end of the third week the operation is interrupted. According to the greater or less demand for oil or tar, the logwood is taken out of the distillation residue from the cooled-down retort, previously broken open in its upper part, and the latter is charged anew with fresh root-stocks, or one closes up the neck of the retort completely, after taking off the dome, in cases when more tar is required, increases the heat of the fire to a considerable extent, and distils the tar straight into an earth cavity by means of a hole struck in the side of the retort, immediately above the hottest place, and with the help of a short, interposed pipe. Out of this cavity the tar is ladled by means of primitive long-handled wooden spoons into casks. In this case the distillation-residue does, of course, not consist of "logwood", but rather of an excellent coke for forging purposes.

In proceeding according to this extremely simple method one obtains a non-homogeneous mixture of acid water and light oil, of amber- to nut-coloured, more or less resinous pine oil, and of thin tar which contains first of all a considerable amount of oil that, however, rapidly diminishes.

In a large plant, visited by Lange, more practical methods were in use; the oil was allowed to settle in large glass bottles in full daylight and the somewhat bleached oils united, after their being separated from water, according to colour and transparency; the tar yield in these works was also separated into 3 portions of varying viscosity.

The manufacture of cart-grease is always combined with that of pine oil, as it finds a ready market in every direction, whereas the distillation-products have to be sent a long distance for the purpose of refining and working up, even abroad.

The most expensive part of the whole operation is the digging-up and preparation of the root-stocks, as one charge of the retort, capable of receiving about 100 cbm., costs 150 to 180 roubles. The distillation of 50 cbm. of wood, having a weight of about 3500 kg. and requiring 12 to 15 cbm. of fuel (either poplar or pine wood),

produces a yield of 700 to 1000 kg. of pine oil, 1000 kg. of wood tar, and 4800 kg. of charcoal, suitable for forging purposes.

H. Harkort[1]) has also described the production of oil of turpentine, or rather of pine oil, tar, and charcoal in Poland. He publishes his remarks as a sort of continuation of Schelenz's[2]) paper on the same subject which we have also discussed; however, we did not give a description of the distillation plant because, as Harkort also remarks, it appears to have been defective and incomplete, probably owing to the fact that Schelenz had to depend for his information on facts adduced by other people. Harkort was in a position to rectify Schelenz's data, as he was able to study such a plant in action himself and obtained reliable information as to the construction and dissemination of the individual types of heating apparatus.

As a matter of fact, it is a question in such works to produce "oil of turpentine", (pine oil), tar, and charcoal from the root-stocks of Scotch pines. These root-stocks ought to remain in the ground as long as possible after the trees have been felled, even for 10 or 15 years, as the yield of pine oil is said to depend mainly on this circumstance. Only in the case of the Bialovitsh forest the root-stocks are said to be so full of oil that they can be utilized even after only 2 years. The outside layers of the root-stocks are, of course, quite rotten then and must be removed by being hacked off. The dry distillation process is conducted according to 2 different methods, either in "Polish" or in "Russian" heating plants.

The Polish furnace is the older system. It is a case of a round furnace whose utmost dimensions do not exceed 4.20 m. in diameter and 4.20 m. in height, with a lower, almost perpendicularly rising part, and a dome. The former is built up of a wall of a thickness of 3 bricks and rises to half the height of the total elevation, whereas the dome is only one stone thick. At both sides of the working-door which is placed quite low down, the fire-places are fixed whose fire-gases pass through horizontal channels, are conducted on the opposite part of the furnace side by means of the perpendicularly rising conduits to a higher level, and lead back by means of a second perpendicular conduit on both sides, ultimately reaching the chimney which is about above the working-door. The floor of the furnace is provided in the middle with a gradient, thereby permitting the collecting tar to find an exit outside through a channel. After charging the plant with root-stocks the furnace is lighted, wood being used. Pine oil, mixed with water-vapour, then passes over for 3 or 4 days, and in point of fact through a lateral opening in the dome or better still through one at the apex, to which a condensation tube is affixed. After this period the tar begins to run along the floor of the furnace, the upper distillation opening being then closed. The heating process is continued till no more tar runs off. By that time the contents of the furnace have been converted into charcoal.

This process is a kind of dry distillation, whereby the production of "oil of turpentine" in the real sense of the word, *i. e.* of a product resulting from gum turpentine by distillation with steam, is quite excluded, on the contrary merely a kind of pine oil is produced. Even the oil which first distils from a "Polish furnace" is quite impure and the yield suffers from the circumstance that a too early rise of temperature decomposes the turpentine still present. During the period of pine oil production the temperature ought not to rise above 100°, in spite of this fact, however, after 3 or 4 days this increase takes place, so that the condensation-products are so dark in

[1]) *Zeitschr. f. angew. Chem.* **29** (1916), I. 361. — [2]) *Ibidem* 251; *Report* October **1916**, 55.

colour, being so much contaminated by tarry decomposition-products, that the collection of pine oil[1]) is interrupted.

An improvement of this process ought to be feasible by carefully keeping to a lower temperature for some time. With this object in view a new furnace construction was introduced, the so-called "Russian furnace". This type was probably first built in the Bialovitsh district about 20 years ago and then transplanted into other regions. It only has one furnace which distributes its gases right and left into conduits which are also placed horizontally. The latter then rise at the opposite side of the furnace and are conducted backwards and forwards 3 times, so that 4 ring-shaped conduits lie in the lower, more stoutly built portion of the furnace. These constructions were of the same size as with the "Polish furnace". It was only at a later period that much larger ones were built, up to a diameter and height of 5.60 m., and at the same time the firing was altered in such a manner that now only one horizontal channel existed, and that the gases were conducted up and down in a zigzagging fashion on both sides back to the chimney which is placed close to the fire-place. The dome, of the thickness of one brick, has an aperture at the top which is covered by a copper head into which two copper elbows of about 20 cm. diameter are inserted. These elbows terminate in tubes, each of 4 m. length (formerly of copper, now made of wood) and finally in a coil condenser with 4 spirals. The cooling operation is performed in a tank filled with water, and the condensation-products run out of the mouth of the cooling coil into a cask let into the earth, in which the oil and water are separated and even the tar which has been carried over is precipitated on the bottom. The condensed water is carried off by means of an overflow pipe and the oil, floating on the surface, poured off into glass bottles. As the end of the wooden conduit is another outlet, out of which the tar which has been condensed here runs off. The lower, stouter portion of the furnace is partly built into the earth, with a view to better isolation, and earth is likewise accumulated round it up to the point where the dome begins.

In this method of working the production of pine oil is absolutely separated from that of tar and charcoal.

The "Polish furnace" serves the purpose of obtaining tar and charcoal of sterling value and the best possible yield, whereas the "Russian system" produces the most pure "oil of turpentine", in as large quantities as possible. This notion is absolutely confirmed by the fact that the plants in the Bialovitsh district, in whose establishments frequently both furnace systems are represented, only run their "Polish furnaces" when tar and charcoal are much in demand; however, they start their "Russian" ones when pine oil is called for which is generally the case. For this reason the more modern works are only fitted up with "Russian furnaces".

The works in the Minsk Government are said to have been erected according to more modern methods. The dome of the furnaces consists of sheet iron the lower part of which is immured into the wall of the lower furnace jacket. The distillation of the pine oil in these furnaces is said to take much less time which is explained by the fact that, owing to the dome getting hot quicker, the mixture of the pine oil

[1]) In this paper as well as in most other publications on turpentine products, the terms "Turpentine" and "Oil of Turpentine" are mixed up and misapplied. If this is the case in ordinary intercourse, and for instance a druggist sells "oil of turpentine", when "turpentine" is demanded, these terms ought certainly not to be confounded in scientific papers and the terms "Turpentine" (*i. e.* the original gum resin), and "oil of turpentine" (*i. e.* the oil produced from turpentine by distillation with steam), ought to be clearly divided. The same objectionable practice is paramount in English and especially in American publications. In the latter case even the terms "Rosin" (colophony), and "Résin" (turpentine) are confounded. In the paper we are discussing here, oil of turpentine is mixed up with pine oil.

and water vapour does not condense and flow back so long as when a dome, made of stone, is employed which of course remains cool much longer.

Harkort had the opportunity of closely investigating such a plant with Russian furnaces, combined with the production of tar, which enables him to give particulars concerning the method of working, yield, &c.

It was a case of a plant consisting of three furnaces: a smaller one of 4.20 m. height and diameter, with 4 ring-shaped fire-conduits, and of 2 larger ones of 5.60 m. height and diameter, with the zigzagging arrangement mentioned above. The furnaces are in such a position that their fire-places are protected by a covered shed. The chimneys have the same height as the furnaces themselves and are built up, close to the fire-place, on the stout side of the lower part of the furnace. The outside dimensions of the chimneys are about 40 × 80 cm. They contain 2 flues, corresponding to the division of the fire left and right. The furnace is charged with root-stocks by means of an aperture on the floor and another one in the dome. The small furnace, having a calculated available space of about 39 cbm., is charged with 32 cbm. of root-stocks which produce about 700 kg. of pine oil during one heating period of the furnace, lasting 15 to 16 days. The dimensions of the large furnace are about 90 cbm. It is charged with 72 cbm. producing about 14 kg. of oil. When one connecting conduit is used the time of charring amounts to about 20 days, when 2 are employed, about 16 days. The heating material which is not placed on a grate consists of wood or bark, and first of all the fire is actively fanned. After 3 or 4 days the first traces of oil appear at the end of the cooling pipe in an almost colourless, transparent state. Day by day it becomes yellower in colour, whereby the fire is reduced and finally is only allowed to glow. The termination of the process is announced by the appearance of a very dark oil which is collected separately, whereas the distillation-products of all the previous days are united. The dark oil can be made clear by standing for a long time. After the fire is extinguished the draught in the chimney is closed by means of two dampers with the object of recharging the furnace in as warm a state as possible. If the furnace is allowed to get quite cold, the yield of the oil is said to diminish considerably. The consumption of firewood amounts to about 40 cbm. = 22500 kg. for one charge of the large furnace. Even 2 days after the fire has gone out oil still continues to pass over, after which the apertures in the head are closed with stoppers.

Doubtless the successful development of the furnace system proves that one has tried to improve it, and the method of distillation employed can by no means be described as primitive and medieval. From the technical point of view the construction of the furnace is at least on a level of many of our own ceramic plants working with periodically heated furnaces.

Harkort asserts the following concerning the dissemination of such plants in the occupied districts: —

In the Government of Grodno, 22 are to be found, of which 9 exist in the forest of Bialovitch. In the Government of Wilna 7, in that of Minsk 3, and in that of Courland 4. Only part of them have been re-started, several having been completely destroyed. On the whole 36 plants with about 90 furnaces and an average production each of 120000 kg. (12000?) of pine oil a year or with a total yearly production of 1100000 kg. are in existence. If one calculates the price of one kg. of pine oil at 3 marks (decidedly a war-price, the price in times of peace only amounting to about 40 pf.), the sum total would be about 3300000 marks. Doubtless this production could be considerably raised by means of an extensive and systematic increase of the plants.

Wormseed Oil, American. The samples of American wormseed oil which came to hand in the course of the last yearly reporting period were again invariably quite inferior substitutes which mostly did not contain even a trace of genuine chenopodium oil. We should only have to repeat what we said in our two last *Reports* if we were to discuss them in detail here, and we confine ourselves to the remark that besides the substances already mentioned formerly as components of these substitutes: eucalyptol, anethol, and menthol, recently also an ester, apparently amyl acetate is employed. There is one case, however, which we should not like to pass over, because the impudence with which the manufacturer stands up for his product, deserves to be more widely known.

Under the designation *Ol. chenopodii artific.* an oil was sent us for investigation which gave the following constants: $d_{15°}$ 0.9536; α_D — 3° 18′; ester value 156.8; soluble in 1.7 vols. of 70 per cent. and 0.5 vols. of 80 per cent. alcohol, showing strong opalescence in each case on more of the solvent being added. Of these constants, the exceedingly high ester value is surprising, as chenopodium oil only contains merely traces of ester compounds. Even if in an artificial product not all the qualities of the natural oil were expected to be present, such a decided difference as the one mentioned above, immediately proves that the composition of this substance must be a very different one. On further examination we found that the characteristic components of the chenopodium oil were totally absent in this oil and that in the main it consisted of compounds which in no case occur in the genuine oil, namely of eucalyptol, anethol, and of an ester, probably amyl acetate. Unfortunately, the nature of this ester could not be exactly determined, as only a small quantity was available. Of course, this compound differed completely in smell and taste from genuine chenopodium oil and it was self-evident that under these circumstances its effect was quite different. Such a fabrication can by no means be termed a substitute for chenopodium oil and accordingly the designation *Ol. Chenopodii artific.* is decidedly inadmissible, as one demands from such a product that at least it should contain the essential and in this case indispensable component of chenopodium oil, namely, ascaridol, as otherwise one might designate any other oil, to which anthelmintic qualities are ascribed, for instance tansy oil, as *Ol. Chenopodii artific.* and this would certainly be going too far. All attempts to produce an artificial chenopodium oil are doomed to be in vain, till one has succeeded in producing ascaridol, and there is no prospect of this at present.

It is perfectly comical to see the manner the manufacturer tries to defend his product. Amongst other statements, he says that the main component of the chenopodium oil is eucalyptol and that this substance is also present in his artificial oil in a corresponding ratio. He further adds that as an artificial compound it could not be a complete substitute for genuine oil, but that it was just as harmless as the latter.

In considering the opinion stated as to the composition of the oil one must ask oneself involuntarily if the manufacturer intended to lead his clients astray consciously or if he has not the faintest idea what *are* the components of chenopodium oil. His saying that eucalyptol is present in his oil "in corresponding ratio" sounds rather suspicious, but in spite of that, we will give him credit that he knows nothing whatever about the matter, and we regard it as deplorable that the manufacture of an artificial chenopodium oil should have been attempted by such an ignoramus. The negative results of his proceedings became patent.

Concerning its harmlessness his product would certainly surpass the genuine oil, as we can assure the manufacturer for the sake of his peace of mind, since the genuine

product is undoubtedly by no means harmless, as experiments have proved[1]), even if from a therapeutic standpoint it can be regarded as non-poisonous.

The manufacturer's confession that his product was not intended to be of equal money value as the genuine oil, was also a point of interest to us, because to judge from another of his letters he maintained the point of view that the effect of his oil was the same as that of the genuine product, which comes to the same as saying that it was a complete substitute for the genuine article. Our expert opinion was probably the cause that the gentleman became more modest in his views.

An essay by P. Trendelenburg[2]) entitled, "On the effect of santonin and its derivatives on the muscles of worms, and remarks on the effect of *Oleum chenopodii*" contains, besides a description of the effect of santonin and its derivatives on the muscles of worms, the frog's heart, and the nervous system of mammalia, some particulars on the effect of American wormseed oil[3]). He even succeeded in depicting the effect graphically. However, a description of his method (which bases in the main on the isolation of the muscular system of the worms) would take up too much of our available space, hence for particulars we must refer the reader to the original paper. The paralyzing effect of chenopodium oil is not of a central nature, as Trendelenburg was able to prove on an isolated preparation of an ascaris muscle, deprived of its nerves; even in that case the isolated strip of muscle is paralyzed by the chenopodium oil and quickly loses its flexibility. Rain-worms which were immersed in an emulsion of chenopodium oil were just as easily paralyzed as the preparation of the ascaris muscle. A solution of 1:500 paralyzed them in 5 minutes; in the ratio of 1:1000, in the space of $^1/_2$ to $^3/_4$ of an hour; in the ratio of 1:4000, in about 2 hours; a solution in the ratio of 1:8000 only leads to a reduction of their movements, whereas a solution in the ratio of 1:16000 not only has no paralyzing effect, but acts as a stimulant.

It was further possible to show in the experiments on the muscles of worms that the stimulating effect which preceeds the paralysis is also peripheral in nature. This stimulating effect in strong concentration (in the ratio of 1:1000) is just as strong as in concentrated solutions of santonin, but it only lasts a very short time; in a few minutes the muscle is completely paralyzed, with reduction of the tonus.

In weaker concentrations, in the ratio of 1:10000 to 1:50000, which latter neither paralyze the whole worm nor the isolated strip of muscle, the muscular system is strongly stimulated for many hours, whereby curves are obtained which are equal to those produced by santonin.

Accordingly, the effect of chenopodium oil on the stimulation of the muscles of worms is dependant on the use of weak concentrations, as in the case of santonin, paralysis following on the use of stronger ones.

Wormwood Oil. Our Miltitz fields have supplied us abundantly with an excellent oil, owing to which we can afford to dispense with the unavailable American shipments. The area planted with wormwood can be extended according to the demand, so that there is no fear of any scarcity.

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[1]) Comp. *Report* April **1907**, 102; October **1911**, 97; October **1912**, 113; October **1915**, 42; October **1916**, 61. — [2]) *Arch. f. exper. Pathol. u. Pharmakol.* **79** (1915), 190. According to a separate copy kindly forwarded to us. — [3]) Comp. *Report* October **1916**, 61.

Pharmacopœias.

American Pharmacopœia.

(The Pharmacopœia of the U. S. of America, 9th decennial revision.)

The 9th edition of the American Pharmacopœia, which became operative on Sept. 1st 1916, unfortunately only reached us in the course of this year owing to war conditions, so we had no time to discuss it in this *Report*, which is in so far a matter of no importance, as all communications with the U. S. are interrupted. We shall remedy this omission in our next *Report*.

Supplement to the Pharmacopœia of the German Empire.

4th Edition.

We have not only undertaken the task of discussing at this point the articles referring to our branch of industry, contained in the new editions of pharmacopœias, but besides we have extended this duty towards publications of a similar nature. To this class belongs the Supplement of the *Ph. Germ., ed. V,* whose third edition we also criticized from that point of view[1]). The 4th edition of the Supplement that appeared last year is now the object of our remarks.

In distinction to the 3rd edition, several articles have undergone changes. The following have been left out altogether: valerian oil, oil of bitter almonds, pine needle oil, melissa oil and Ceylon cinnamon oil which has become official. The following have been newly added: carvone, oil of cassia, cubeb oil, cypress oil, eugenol, Siberian pine needle oil, helenine, heliotropine, marjoram oil, menthyl valerate, *iso*amyl salicylate, and American wormseed oil.

One can in a general manner declare oneself satisfied with the requirements applied to the various oils, &c. which have been enlarged by adding data concerning rotation, in distinction to the former ones; one notices that the criticisms directed against the former edition have not been passed over, and besides, that the latest literature has been consulted. In spite of this, of course, something is left to be desired, as will be apparent from the discussion which now follows.

The rotation constants invariably refer to an observation temperature of 20°. The strength of the spirits of wine corresponds to the demands of the *Ph. Germ., ed. V, viz.,* spirits of wine with 90.09 to 91.29 per cent. by volume and diluted spirits of wine with 68 to 69 per cent. by volume.

Amber Oil, rectified *(Oleum succini rectificatum).* Light-yellow, brownish-yellow, or olive-coloured; $d_{15°}$ 0.920 to 0.935[1]); α_D + 22 to + 31°; soluble in 4 to 5 vols. of 95 per cent. alcohol[2]).

[1]) During recent years, we have observed, in our own distillates, specific gravities up to 0.949.

[2]) Up to 7 vols. are in some cases required in order to effect a complete solution.

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[1]) *Report* October **1906**, 92.

***iso*-Amyl salicylate** *(Amylium salicylicum)*. Colourless to yellowish; insoluble in water, easily soluble in chloroform and ether, also in 3 parts of alcohol; boiling point about 290°[1]); $d_{15°}$ 1.049 to 1.056; proof of identity by means of ferric chloride.

[1]) Our own preparations gave a boiling point of 276 to 277° (743 mm.).

Anethole *(Anetholum)*. White crystalline mass, smelling of aniseed oil and tasting exceedingly sweet, melting to a colourless, optically inactive, very refractive liquid; boiling point 232 to 234°; melting point 22 to 23°[1]); $d_{25°}$ 0.984 to 0.986.

[1]) In compounds melting at such a low temperature, it is preferable to determine the solidification point which is between 21 and 22°, if the liquid is cooled down to + 16°.

Bergamot Oil *(Oleum bergamottæ)*. Greenish-yellow to green in colour, occasionally also honey-coloured; $d_{15°}$ 0.881 to 0.886; α_D + 8 to + 20°[1]); soluble in 0.25 to 0.5 volumes of spirits of wine, the solution must not become turbid on adding more spirits of wine[2]); evaporation-residue at the utmost 6 per cent.; linalyl acetate content at least 36 per cent.[3]).

[1]) The upper limit is too low, it ought to be 22°.

[2]) A slight opaqueness is often observable in otherwise quite first-rate oils.

[3]) Oils with the demanded minimum ester content are not always obtainable, so that one often has to be satisfied with 34 per cent. linalyl acetate.

Cajuput Oil, rectified *(Oleum Cajuputi rectificatum)*. Colourless to yellow; constants; $d_{15°}$ 0.915 to 0.930; α_D lævorotation, up to − 4°; soluble in any quantity of alcohol; test for absence of copper; the crude oil contains copper, but otherwise shows the same properties as the rectified product.

Carvone *(Carvonum)*. Colourless or light yellowish; boiling point 229 to 230°; $d_{15°}$ not below 0.960[1]); α_D + 50 to + 62°; soluble in two parts of diluted alcohol.

[1]) One ought to demand a specific gravity not less than 0.963.

Cassia Oil *(Oleum Cinnamomi Cassiæ)*. Yellow to brownish; $d_{15°}$ 1.055 to 1.070; soluble in 3 parts of diluted alcohol, giving a clear or only slightly opalescent solution, and soluble also in every quantity of spirit. Residue on evaporation, when one gram is evaporated, at the utmost only 8 per cent.[1]); cinnamaldehyde content (determined by the sodium bisulphite method) at least 74 per cent.[2]).

[1]) Cassia oil only evaporates slowly because it consists mainly of high boiling constituents, accordingly it must remain a *very long* time on the water-bath, otherwise the evaporation is not complete and, in consequence, the results are too high. The distillation-test, described in detail in Gildemeister's text-book (2nd ed., Vol. II, page 440) on Volatile Oils, is to be preferred owing to the difficult volatility of cassia oil.

[2]) A fair commercial product contains 80 per cent. of aldehyde and more.

Chamomile Oil, Essential *(Oleum Chamomillæ æthereum)*. Viscous, at low temperatures having almost the consistency of butter; intense dark blue which colour gradually changes to green and brown under the influence of light and air; $d_{15°}$ 0.925 to 0.940[1]).

[1]) These limits ought to be increased in both directions.

Coumarine *(Cumarinum)*. Colourless, glistening, sublimable prisms which dissolve with difficulty in cold water, easier in boiling do; easily soluble in alcohol and ether and in warm caustic soda. Melting point about 70°; combustion-residue at the utmost 1 per cent.; test for absence of acetanilide.

Cubeb Oil *(Oleum Cubebarum)*. Occasionally colourless, mostly light-green to blue-green: $d_{15°}$ 0.915 to 0.930; α_D − 25 to − 43°.

Cypress Oil *(Oleum Cupressi)*. Yellowish; $d_{15°}$ 0.868 to 0.900; α_D + 4 to + 31°.

Eucalyptole *(Eucalyptolum).* Colourless; boiling point 176 to 177°; congelation point about 0°; $d_{15°}$ 0.928 to 0.930; $\alpha_D \pm 0°$; very little soluble in water, mixes with alcohol, ether, chloroform, and fatty oils; equal volumes of eucalyptole and liquid paraffin ought to give a perfectly transparent mixture.

Eugenol *(Eugenolum).* Light-yellow, getting brown on exposure to the air; $d_{15°}$ 1.071 to 1.074; $\alpha_D \pm 0°$; soluble in two parts of dilute alcohol; 1 gram of eugenol ought to give, with 26 ccm. of water and 4 ccm. of caustic soda, a clear liquid which becomes slightly turbid on exposure to the air; identifiable by adding ferric chloride.

Helenine *(Heleninum album).* Colourless, needle-shaped crystals, almost insoluble in water, easily soluble in alcohol and ether. Melting point 72 to 73°; combustion-residue at the utmost 1 per cent.; the alcoholic solution (1 + 24) must not redden wet litmus paper; 5 ccm. of this solution must not become blue or green on addition of one drop of ferric chloride solution, nor may they be rendered turbid by adding one drop of iodine-potassium iodide solution.

Heliotropine *(Heliotropinum).* Colourless, glistening crystals, hardly soluble in water, but easily in alcohol and ether. Melting point 37°[1]); combustion-residue at the utmost 0.1 per cent.; 0.1 gram. of heliotropine ought to give with 2 ccm. of sulphuric accid a lemon-coloured liquid, by no means a brown-coloured one. 5 ccm. of an alcoholic solution (1 + 24) ought to give a pure yellow colour on adding one drop of ferric chloride solution; test for absence of acetanilide.

[1]) According to our own observations, this compound melts between 35 and 36°.

Marjoram Oil *(Oleum Majoranæ).* Yellow to yellowish green; $d_{15°}$ 0.890 to 0.910; α_D + 5 to + 18°[1]).

[1]) The upper limit is + 19°.

Menthyl valerate *(Mentholum valerianicum), Validol.* A solution of about one part of menthol in two parts of menthyl*iso*valerate[1]). Colourless; $d_{15°}$ 0.902 to 0.908; scarcely soluble in water, easily in alcohol, ether, and chloroform. Proofs of identity, test for free valeric and hydrochloric acids. 0.2 gram. of menthyl valerate must completely volatilise on the water-bath. Ester value, 155 to 170 (for the purpose of saponification 2 gram. of ester are heated with 30 ccm. of semi-normal caustic potash 5 hours on a wire net, so that the mixture boils briskly)[2]).

[1]) Our own preparation does not consist of the above-mentioned mixture, but is in fact the pure ester.
[2]) Concerning the saponification of menthyl valerate, *vide* page 89 of this *Report*.

Methyl salicylate *(Methylum salicylicum).* Colourless or slightly yellow; boiling point 218 to 221°[1]); $d_{15°}$ 1.182 to 1.189[2]); little soluble in water, very easily in alcohol and ether, mixes with fatty and essential oils; proof of identity by means of ferric chloride.

[1]) The boiling point occasionally rises to 222 to 223°.
[2]) The specific gravity lies between 1.185 and 1.190.

Neroli Oil *(Oleum Aurantii Florum).* Yellowish to brownish; $d_{15°}$ 0.870 to 0.880[1]); soluble in 1 to 2 vols. of alcohol; the solution shows a violet-blue fluorescence[2]).

[1]) The upper limit ought to be extended to 0.881.
[2]) The ratio of solubility mentioned holds good already for alcohol of 80 per cent. by volume.

Orange Oil, bitter *(Oleum Aurantii Corticis).* Yellow to brownish; $d_{15°}$ 0.852 to 0.857; α_D + 88 to + 98°[1]).

[1]) The rotation only reaches + 98° in the case of *sweet* orange oil, in the case of the *bitter* compound it only reaches + 94°.

Origanum Oil, Cretian *(Oleum Origani cretici)*. Rather viscous; yellow to red-brown; $d_{15°}$ 0.920 to 0.980; soluble in 3 parts of dilute alcohol. Phenol content at least 60 per cent. (determined with 5 per cent. caustic soda solution).

Parsley Seed Oil *(Oleum Petroselini)*. Rather viscous; yellowish to light green; $d_{15°}$ 1.050 to 1.100[1]).

[1]) The limits mentioned do not quite tally with the real ones, the values ought to be 1.03 to 1.01.

Pine Needle Oil, Siberian *(Oleum Pini sibiricum)*. Colourless to light yellowish-green; $d_{15°}$ 0,905 to 0.925; α_D — 37 to — 43°; soluble in 0.5 to 1 vol. of alcohol, giving a clear solution or showing only slight opalescence.

Oil of Pinus montana, Mill. *(Oleum Pini Pumilionis)*. Colourless to light yellow; $d_{15°}$ 0.860 to 0.875[1]); α_D — 4.5 to — 15.5°; soluble in 5 to 7 volumes of alcohol, giving a clear, or at least an only slightly turbid solution[2]). Not more than 10 per cent. ought to pass over under 165° on fractional distillation.

[1]) In Tirolese oils, we have observed as low values as 0.858.

[2]) Under certain conditions, up to 8 vols. of alcohol are required to effect complete solution.

Rue Oil *(Oleum Rutae)*. Colourless to yellow, generally fluorescent; $d_{15°}$ 0.833 to 0.847; soluble in 2 to 4 vols. of dilute alcohol, occasionally with opalescence.

[1]) The condition demanded in the previous edition of a fixed congelation-point has been dropped in the present one, owing to the variable composition of various oils of rue.

Sage Oil *(Oleum Salviæ)*. Yellowish to greenish-yellow; $d_{15°}$ 0.915 to 0.930; α_D + 9 to 25°[1]); soluble in every volume of alcohol.

[1]) The lower limit must be reduced by + 2°.

Sassafras Oil *(Oleum Sassafras)*. Yellow to reddish-yellow: $d_{15°}$ 1.070 to 1.080; α_D + 2 to + 4°; soluble in 1 to 2 vols. of alcohol.

Savin Oil *(Oleum Sabinæ)*. Colourless to yellowish; $d_{15°}$ 0.907 to 0.930; α_D + 38 to + 62°; soluble in 0.5 vols. and more of alcohol.

Spearmint Oil *(Oleum Menthæ crispæ)*. Colourless, or yellowish to greenish-yellow; $d_{15°}$ 0.920 to 0.940; α_D — 35 to — 50°[1]); soluble in 1 to 1.5 parts of a mixture of equal volumes of alcohol and diluted alcohol (to about 80 per cent. by volume); the solution becomes turbid[2]) on further dilution.

[1]) The limits ought to be extended to about — 34 to — 52°.

[2]) The right expression would be: opalescent to turbid.

Spike Oil *(Oleum Spicæ)*. Yellowish; $d_{15°}$ 0.905 to 0.915°[1]); soluble in 2 to 3 parts of dilute alcohol, giving a clear solution or at least one that is only slightly turbid[2]).

[1]) The upper limit must be extended to 0.918.

[2]) The solution must only be opalescent in the very *least* degree.

Star Anise Oil *(Oleum Anisi stellati)*. Colourless or yellowish; $d_{20°}$ 0.980 to 0.990; congelation-point not below 14°[1]); soluble in 1.5 to 3 vols. of alcohol.

[1]) In this case, the oil must be cooled down to + 10°; in some cases congelation is started by adding a trace of solid anethol or of oil of anise.

Tansy Oil *(Oleum Tanaceti)*. Yellow to greenish-yellow, becoming brownish under the influence of light and air; $d_{15°}$ 0.923 to 0.940[1]); easily soluble in alcohol.

[1]) In the 3rd edition of the supplement, the upper limit was mentioned more correctly as being 0.954, since with oils produced from the dry herb values reaching 0.955 are obtained.

Wild Thyme Oil *(Oleum Serpylli).* Colourless to golden-yellow; $d_{15°}$ 0.890 to 0.920; α_D — 10 to — 20°[1]); soluble in every volume of alcohol.

[1]) Even — 21° have been observed.

Vanilline *(Vanillinum).* Fine crystal-needles; soluble in 80 to 100 parts of water of 15° and in 20 parts of 75 to 80°; easily soluble in alcohol, ether, and chloroform; melting point 81 to 82°; combustion-residue at the utmost 0.1 per cent.; the aqueous and alcoholic solution of vanilline turns blue on adding ferric chloride solution; 0.1 g. of vanilline ought to dissolve in 2 ccm. of sulphuric acid, when heated gently, giving a clear solution of a light yellow colour and without leaving any residue.

Wintergreen Oil *(Oleum Gaultheriæ).* Colourless, yellowish or reddish; $d_{15°}$ 1.180 to 1.193; $\alpha_D \pm 0°$ or slightly lævorotatory, up to — 1°; soluble in 5 to 8 vols. of dilute alcohol; 1 ccm. of wintergreen oil ought, on mixing with 10 ccm. of a 5 per cent. caustic potash solution, to give a colourless or at least only slightly yellowish solution; test for identification by means of ferric chloride.

Wintergreen Oil, artificial, see Methyl salicylate.

Wormseed Oil, American *(Oleum Chenopodii anthelmintici).* Colourless to yellowish; $d_{15°}$ 0.965 to 0.990 and higher; α_D — 4 to — 8.9°; soluble in 3 to 10 vols. of 70 per cent. alcohol.

Wormwood Oil *(Oleum Absinthii).* Rather viscous; generally of a dark-green colour, occasionally also blue or brown; $d_{15°}$ 0.900 to 0.955.

In a discussion of the *Ph. G. ed. V,* G. Frerichs[1]) also refers to the estimation of oil of cinnamon, for which purpose the Pharmacopœia requires a sodium bisulphite solution of about 30 per cent. According to Frerichs, it would be practical to add that the solution must be prepared afresh in each case and should also be filtered. Commercial sodium bisulphite is mostly lumpy and only dissolves very slowly in cold water; accordingly, it is advisable to pound it in a mortar beforehand. The solution ought in no case to be heated. About 60 g. of the solution are required for every determination. The best way is to dissolve 25 g. of powdered sodium bisulphite in 45 g. of cold water, in which manner one can easily obtain the necessary quantity of filtered solution.

In carrying out the determination, it is imperative to shake the solution during the heating process frequently and steadily. When the undissolved oil has been brought into the neck of the little flask, the latter must still be heated for some time, being kept submerged half-way up the neck. Observations ought only to be noted after the apparatus has cooled down. The neck of the little flask ought to be adjusted for practical purposes in such a manner that the 50 ccm. mark is in the middle of the neck, from which point the latter ought to be divided into $^1/_{10}$ ccm. to a length of 52 ccm. upwards and 48 ccm. downwards.

In carrying out the determination, the aqueous liquid ought to be filled up to the 50 ccm. mark during the heating process, because under these circumstances the undissolved oil is to be found within the scale after cooling down. The interior diameter of the neck may be about 10 mm. The little flask can also be used for other purposes as a measuring flask. The graduation of the neck, just mentioned, renders it also suitable for testing oil of thyme.

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[1]) *Apotheker Ztg.* **32** (1917), 341, 347.

The Pharmacopœia prescribes the following method of standardization for this oil:— if one shakes 5 ccm. of oil of thyme thoroughly with a mixture of 10 ccm. of caustic soda solution and 20 ccm. of water and than lets it stand till the layer of caustic soda has become clear again, the volume of the oil-layer swimming on its surface must not exceed 4 ccm. at the utmost.

Frerichs adds the following remarks:— that the determination, if executed with a graduated measuring cylinder of 100 ccm. capacity, is always very inexact. Further, that the little measuring flask used for the standardization of oil of cinnamon is very suitable for this purpose, if the neck is graduated for at least a space of 4 ccm. By filling up with the same mixture of caustic soda and water, the undissolved oil can be brought up to the upper mark. The aqueous liquid only becomes clear after a very long time. The oil itself remains turbid even for a much longer period. However, it is sufficient to let the little flask stand just long enough for the oil to separate from the aqueous liquid.

Chemical Preparations and Drugs.

Almond Oil, expressed. The present lack of fatty almond oil and of other oils and fats has also drawn general attention to the utilization of plum stones[1]). Hence F. Darvas[2]) publishes that he produced a first-rate table oil from plum stones. For trial purposes he freed 5 kg. of plum stones from their outside hard shells and thereby obtained 1 kg. of kernels, from which he produced 200 g. of fatty oil. He submitted the expressed residue to dry steam distillation and collected the distillate in 20 per cent. alcohol, whereby he obtained 670 g. of a solution of 1.3 to 1.4 per cent. benzaldehyde cyanhydrine. The residue consisted of a press-cake, poorer in oil, but richer in albumen, weighing about 800 g. On repeating his experiments the results hardly varied at all.

The fatty plum oil *(Oleum prunorum)* had, after necessarily being purified, a golden-yellow colour, was transparent, totally odourless, had a pleasent mild taste and a congelation point of about —17°. Constants, d 0.9169 to 0.918; acid value 1.8 to 2.1; iodine value, 97.5 to 100.6. One can say in a general manner that plum oil ranks between almond oil and sesame oil; it is equally suitable as a table oil and for medical purposes.

The *Aqua prunorum* prepared by Darvas according to the prescription of the Hungarian Pharmacopœia for the preparation of *Aqua amygdalarum amararum* was a solution of from 1.3 to 1.4 per cent. benzaldehyde cyanhydrine. The distillate obtained was diluted to a content of $^1/_{10}$ per cent. The *Aqua prunorum,* thus prepared, is colourless and quite transparent; the smell is exactly like a good quality of bitter almond water. The residue of the preparation of plum oil and of plum water can be used for cosmetic purposes (instead of almond-powder) and also, owing to its richness in albuminous substances, as a particularly nourishing food for animals.

Anethole. According to R. Meldrum[3]), anethole is a highly suitable substance for determining the melting and congelation point, because it crystallizes easily and melts already at ordinary room-temperature. He made his experiments in a tube of about

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[1]) Comp. *Report* October **1916**, 67. — [2]) *Zeitschr. d. allg. österr. Apoth. Ver.* **54** (1916), 419. — [3]) *Chem. News* **112** (1915), 259. — Comp. *ibidem* **111** (1915), 95.

1.9 cm. diameter and in point of fact determined the *melting* point by the following methods:—

1. According to the thermometer-bulb method.

The thermometer is cooled down to zero and is than quickly dipped into liquid anethole of 12°, with the object of forming a coating and to enable the anethole to melt at a temperature of about 16°. Under these conditions, he found the melting point to be about 15.7 to 15.9°.

2. According to the opacity method.

One places the anethole, previously cooled down to +9°, in a water-bath of 14° and raises the temperature every quarter of an hour by 1° while stirring the anethole and water continually at the same time. The temperature at which the liquid becomes quite opaque is then the melting point of anethole. By this means, Meldrum observed a melting point of 17°.

He determined the *congelation*-point by stirring continually in an air-bath of 8 to 12° and by this means observed an average value of 15.36° (greatest divergence 0.6°).

Occasionally, anethole congeals suddenly at 10°, at other times, however, only after having been stirred for 10 minutes; this depends on the temperature at which the solid anethole was liquefied. If the thermometer and the anethole have been heated up to 20° for 10 minutes, all the little crystals floating in the liquid have had time to dissolve, and in consequence the liquid must be cooled down to a considerable extent to enable it to become solid again. On the other hand, anethole which has been liquefied at 17° only congeals at a higher temperature owing to the presence of very finely divided solid particles. The higher the temperature of liquefaction was, the more the liquid must be cooled down. In making the determination on the water-bath, while stirring continually, Meldrum found a congelation-point of 13.8 to 14.7°, on an average 14.1° (divergence 1.2°). The magnitude of the divergence depends on the temperature of the air and water-bath, as is apparent from a table also issued in this paper. Withal, the anethole had been liquefied at a temperature above 18°; if the liquefaction took place at a temperature of 17°, it congealed at about 15.5° (divergence 0.5°). In these last experiments, traces of finely-divided solid substance were present in the liquid.

Hereupon the anethole was liquefied until about 2 per cent. remained solid and then the mass was stirred in an air bath, whereby the congelation point of 14.7° was observed (deviation 0.6°).

In making the determination in a tube of about 2.5 cm. diameter, the congelation point was 14.7° (deviation 0.2°), on using a larger quantity (200 g.) of anethole 14.7°. Even after heating for 20 minutes at 110°, the congelation point of anethole was not modified.

Anethole which had remained for 5 months in sealed glass vessels and had been exposed to light at ordinary room temperature became solid at about 5.5°.

We have only reported these observations of Meldrum in order to show what nonsense is sometimed published, as the paper itself is absolutely valueless. We do not know if it was the fault of the anethole or of the manner in which Meldrum worked, in any case the publication is so involved that one cannot make head or tail of it. We have the impression that the observations must have been made with an exceedingly inferior sort of anethole, yet Meldrum's statement that his purveyor gave the melting point of the anethole as being at 22° is contrary to this, besides which the preparation, as far as Meldrum could determine, was pure. How he controlled its purity is, however, not apparent from his publication.

We will, however, add for Meldrum's benefit that the congelation point of anethole is between 21 and 22°, and the melting point between 22 and 23°. The combined action of air and light reduces the crystallizing capacity of anethole, so that finally it will not crystallize at all. Further particulars on this subject are to be found in Gildemeister's text-book on Volatile Oils, 2nd edition, vol. I, page 478.

Benzaldehyde. When examining a preparation *(benzaldehyd. puriss.)*, bought in Sweden, it struck Wastenson[1]) that during combustion the flame was not so strong as usual. As also the specific gravity at 15° was 0.943 (instead of 1.0504), he examined the preparation carefully, which proved that it consisted of only 20 per cent. benzaldehyde and contained 80 per cent. of alcohol. On mixing 10 ccm. of benzaldehyde with the same quantity of water and agitating with sodium chloride up to saturation the volume of the secreted oil layer amounted to less than 2 ccm.

Such an adulteration of benzaldehyde is, as Wastenson remarks, at present a very profitable business, because the price has risen in the last two years from 10 to 46 crowns for 1 kg.

Benzoin. Formerly, the *Styrax Benzoin*, Dryand., N. O. *Styraceæ*, was regarded as the mother plant of the Siamese gum benzoin. In the year 1912, however, a publication of the Royal Botanic Gardens, Kew, appeared in which it was stated that the gum was produced by another sort of *Styrax, e. g.*, by *S. benzoides*, Craib. This also, however, seems to be incorrect, as H. Rordorf[2]) who has often gone into the question of the origin of benzoin resin now believes as the result of his latest investigations that one must attribute it to a new species which he calls *S. Siamensis*.

From North Siam he obtained fruits of the benzoin tree which he examined carefully and compared with those of *Styrax Benzoin* and of *S. benzoides*, whereby he arrived at the result that they belong to a hitherto unknown species.

He arranged and prepared them in such a manner, that the principal characteristics are made as obvious as possible and then had them photographed; a reproduction of the photograph is to be found as an illustration of his paper.

Bornylisovalerate and Menthylisovalerate. It is notorious that the esters of *iso*valeric acid distinguish themselves by being exceedingly difficult to saponify. Accordingly, it was prescribed for the determination of the ester content of bornyl*iso*valerate and of menthyl*iso*valerate to heat them for *2 hours*[3]) with a great excess of caustic potash, whereby the results for the bornylester amounted to 86 to 89 per cent. and for the menthylester to about 80 per cent. On making investigations for the disclosure of the nature of non-saponified portions of these two esters, we found that even, in spite of observing the saponification conditions just mentioned, the saponification was not complete and that accordingly the above-mentioned results were too low, a fact which applies especially to menthyl*iso*valerate. In order, therefore, to obtain a quite reliable norm for the valuation of these esters, it was primarily necessary to determine in an absolute manner, how great the excess of alkali and the duration of the saponification ought to be in order to *complete* the operation. This investigation showed that for 1.5 g. bornylester a saponification period lasting 3 hours with the aid of 30 ccm.

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[1]) *Svensk farmaceutisk tidskrift* **1916**, 515; *Pharm. Zentralh.* **58** (1917), 51. — [2]) *Schweiz. Apotheker Ztg.* **54** (1916), 585; **52** (1914), 701. — [3]) Comp. Gildemeister and Hoffmann, *The Volatile Oils*, 2nd ed., vol. I, p. 519 and 520.

semi-normal caustic potash sufficed, whereas for 1.5 g. menthylester 60 ccm. semi-normal caustic potash and a saponification period lasting 6 hours were necessary. Under these conditions, bornylvalerate gave about 93 per cent. ester and 6 per cent. free borneol. The free borneol content was calculated from the saponification number increased by acetylation. Menthylvalerate had an ester content of 99 to 100 per cent. and accordingly the saponification number was not raised by acetylation.

The *iso*valeric esters of *o*-cresol and thymol gave after 3 hours' saponification by the aid of 30 ccm. of caustic potash values reaching 99 to 100 per cent.

Accordingly, it is imperative to boil for 3 hours using 30 ccm. of semi-normal caustic potash in order to terminate the operation when using 1.5 g. of *iso*valeric esters and in the case of menthylvalerate 6 hours' boiling and 60 ccm. of caustic liquor are indispensible. When saponifying menthyl*iso*valerate which is so exceedingly stable towards alkali, with 60 ccm. seminormal potash liquor, the following values were obtained in the course of the process:—

after	one	hour	acid value	0,	ester value	149.8	=	64.2	per cent.	ester
„	2	hours	„ „	0,	„ „	190.4	=	81.6	„ „	„
„	3	„	„ „	0,	„ „	210.0	=	90.0	„ „	„
„	4	„	„ „	0,	„ „	218.4	=	93.6	„ „	„
„	5	„	„ „	0,	„ „	229.6	=	98.4	„ „	„
„	6	„	„ „	0,	„ „	232.4	=	99.5	„ „	„
„	8	„	„ „	0,	„ „	233.8	=	100.2	„ „	„

Cinnamaldehyde. P. G. Unna[1]) recommends a mixture of sagrotan[2]) and cinnamaldehyde as a remedy for itch and similar irritation. The scabies-mite is not destroyed by partial sterilisation with sagrotan, on the contrary, in cases in which, besides scabies, eczematic papulæ and small blisters are present and give the false impression of a pseudoscabies, the characteristic appearance of the mite-runs comes fully to light under the sagrotan-application. By the addition of cinnamaldehyde to the sagrotan solution one obtains, however, a solution which acts as an anti-scabietic; it forms a fine golden-yellow, very frothy emulsion which leaves, after application, a thin residue on the skin in which the cinnamaldehyde by contact with the air becomes oxidized to cinnamic acid[3]). By adding small quantities of zinc oxide and chalk, one can greatly increase the anæmising and accordingly anti-itching effect of the the sagrotan and improve the formation of a coating containing cinnamic acid. The prescription runs as follows:—

Cinnamaldehydum	1
Calc. carbon.	10
Zinci oxid.	10
Sagrotan	10
Aq. dest. ad	100

M. f. Lotio Sagrotani contra scabiem.

The mixture only produces a slight burning feeling on the spots that have been scratched. The anti-itching effect commences at once, the mite-killing effect is terminated in 24 to 48 hours, if the compound is applied daily several times on all itching spots.

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[1]) *Dermatol. Wochenschr.* **64** (1914), 234. — [2]) According to the manufacturers, sagrotan is a highly, effective disinfectant like lysol. It is a soap solution which contains as an efficacious component a molecular mixture of chloroxylenol and chlorocresol and is said to have the "double efficacy of both the chemical components added together". (*Prakt. Pharm.* **11** [1914], 44.) — [3]) Cinnamic acid, balsam Peru, storax, benzyl and cinnamyl cinnamates act, according to Unna, as antiscabietics.

The remedy has a pleasant odour of cinnamon, is cleanly and makes hospital-treatment for scabies quite superfluous. Ambulant treatment, added to the patients' coming up for examination every two or 3 days, is quite sufficient.

Eucalyptole. In the present day, catgut is sterilized either by sublimate or by oil of juniper wood and no longer with carbolic acid, as in to Lister's original prescription. According to A. Goris[1]), an even more suitable substance for this purpose is to be found in eucalyptole. It is quite sufficient to leave the commercial preparation in contact with eucalyptole for a few days; however, infected catgut must be treated for a much longer time with eucalyptole. Goris found that juniper wood oil only acts very slowly.

Menthylisovalerate. As to the saponification of this compound, vide p. 89.

Methylbenzoate. Oil of cloves which was introduced into microscopical technique in the year 1865 by Rindfleisch, has been generally appreciated hitherto, and in recent times even more than formerly it proved to be a good solvent for celloidine, wherefore it plays an important part in the double intermedium of celloidine and paraffine which in the present day is much in use. It is not an ideal intermedium, as it has a smell which is disagreeable to some people and also gets very dark in colour in course of time. P. Mayer[2]), therefore, already pointed out in 1891 that in the transference of thick preparations from alcohol into balsam it ought preferably to be removed in advance by xylene, as otherwise it would rapidly become dark therein. Eugenol has the same disadvantages. However, Mayer believes he has found in methylbenzoate a complete substitute in every respect for oil of cloves, adding that one ought to make the following demands on a good substitute:— 1. it ought to be and to remain colourless; 2. it ought not to be dearer than oil of cloves, on the contrary preferably much cheaper; 3. it ought to have a high refractive index, as to enable the objects for observation to be very transparent in it; 4. it ought to mix equally well with 96 per cent. alcohol or even with a weaker product as with xylene or balsam, giving a clear mixture; 5. it ought to dissolve gun cotton easily. Finally, 6. it ought to be regarded as an advantage if it can be produced synthetically, to enable one in buying it not to have to depend on foreign countries. Most of the conditions, enumerated above, are supplied by terpineol[3]) and by benzylalcohol[4]); both are and remain colourless, mix already with 90 per cent. alcohol, euparal and turpentine, the former also with Canada balsam. As they do not dissolve gun cotton at all, they need not be taken into consideration here. Methylbenzoate, on the contrary, meets all requirements. It gives clear solutions with benzylalcohol, xylene and balsam, and without difficulty with 96 or 90 per cent. alcohol, but not with glycerol. It did not dissolve paraffine[5]), but already in one night air-dried celloidine in the ratio of about 14 per cent. to a colourless syrup, even quicker, of course, dry gun cotton, in any case much quicker and more efficiently than is possible with oil of cloves[6]). The thickish solution can be used an intermedium just as well as oil of cloves. The low price is also in favour of methylbenzoate. In this last respect, nitrobenzene would be far preferable, as it also dissolves gun cotton much

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[1]) *Bull. Sciences pharmacol.* **23** (1916), 67. — [2]) *Zeitschr. f. wissenschaftl. Mikrosk. u. f. mikrosk. Technik* **33** (1916), 1. — [3]) Comp. *Report* October **1910**, 153. — [4]) Comp. *Report* April **1915**, 55. — [5]) A considerable amount of paraffine is dissolved by methylbenzoate when left in contact with it for a long period. — [6]) The use of methyl benzoate in microscopical technique is not quite a novelty; it was already recommended, in 1898, by Jordan as Niobe oil. He mentions that it dissolves celloidine well off the sections. Niobe oil, as Mayer himself mentions, to a trade term which was formerly particularly in use for methylbenzoate.

quicker and easier, but it must not be forgotten that its smell is disagreeable and that it is poisonous.

The same applies to benzaldehyde, as it is very cheap, dissolves celloidine very quickly, but has a strong odour and is very rapidly oxidized to benzoic acid. Although it gives a clear field of vision, it would not be possible to employ it even for including preparations temporarily and accordingly one must stick to cedar oil, terpineol, and benzylalcohol. Especially the last-mentioned substance is much to be recommended for this purpose, as it forms so to speak a liquid balsam.

Musk, artificial. *Reduction and hydrolysis of 2,4,6-trinitro-3-*tert.*-butyl-1-methylbenzene and of trinitro-*tert.*-butylxylene.* The part that 2,4,6-trinitro-3-*tert.*-butyl-1-methylbenzene plays as artificial musk in the chemistry of perfumes made it appear desirable to J. Herzig and F. Wenzel[1]) to produce the corresponding phloroglucinol of this compound by reducing and hydrolyzing it and to investigate it thoroughly. The production of the amino-derivate in the form of the hydrochloride succeeded without a hitch; however, on hydrolyzing the amino-derivate the tertiary butyl-group was completely split off, whereby ordinary methyl phloroglucinol was formed. While identifying the latter, the authors observed an interesting dimorphism of the acetyl-product. On first preparing it they obtained a substance melting between 54 and 56°, whereas Weidel[2]) mentions 52°; this product, however, was lost during an analysis which failed, and since that time a compound formed melting, however, at 76°. A methylphloroglucinol also, prepared according to Weidel's directions from trinitrotoluene, produced an acetyl-derivative with the melting point of 76°. However, there appears to be no doubt as to the identity of both substances, so that probably the formation of the acetyl-product with the melting point of 52° will be realized again, but possibly in another laboratory.

Strange to say, when the hydrochloride of the 2,4,6-triamino-*tert.*-butyl-1-methylbenzene is acetylated, one amino-group is exchanged for the acetyl-radicle, an occurrence which finds analogies elsewhere.

It came to light during the experiments with 2,4,6-trinitro-*tert.*-butyl-xylene that also in this case the *tert.*-butyl-group during hydrolysis was split off, so that dimethylphloroglucinol was formed as a final product. Occasionally, the scission already takes place during the reduction, for which reason it is by no means easy to obtain the hydrochloride of the triamino-*tert.*-butylxylene as a pure substance. Quite apart from the separation of the tertiary butyl group, even the amino-groups are substituted by the hydroxyl group owing to partial hydrolysis. It was possible to isolate 3 different compounds from the crude reduction product, namely the hydrochlorides of the triamino-*tert.*-butylxylene, the triaminoxylene, and the diaminohydroxyxylene.

Unfortunately, the experiments offered no certain explanation as to the manner in which the tertiary butyl group was split off during hydrolysis. The possibility of splitting off the alkyl groups seems to depend greatly on their size and ramifaction. The methyl group proved to be absolutely resistant. Doubtless, it will be impossible by means of hydrolysis to produce phloroglucinols with large, especially with ramified alkyl-radicles.

Peru Balsam. What wonderful tricks in the shape of fraudulent proceedings come to light now-a-days were disclosed to us on examining a product called *Cinnameinum album,* which we received for investigation from a Berlin firm. Its colourlessness and

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[1]) *Monatsh. f. Chem.* **37** 1916), 567. — [2]) *Ibidem* **19** (1898), 223.

its odour proved to us that this case was not a question of cinnamein from Peru balsam, we believe at least however to have a mixture of benzylbenzoate and benzylcinnamate before us. This supposition proved on further examination to be erroneous; on the contrary, the preparation consisted in the main of phthalic ester which had been perfumed with the aid of little artificial musk. As only 2 ccm. of the oil were at our disposal, it was not quite easy to prove this and especially it was not possible to determine if the preparation contained other compounds besides those already mentioned. The high ester value (499; ethyl phthalate has an ester value of 504.5), permits of drawing the conclusion that the quantity of such admixtures can only have been quite irrelevant.

Peruol, a substitute for Peruvian balsam, already recommended as such in time of peace, consists of a solution of peruscabin (benzylbenzoate) in castor oil. According to F. Curschmann's[1]) advice, vaseline oil has latterly been substituted for the castor oil without diminishing the effect of the preparation.

Pollantin. W. Gaehtgens[2]) publishes an ample treatise on hay-fever and its treatment, in which he takes into consideration older publications as well as the most recent investigations. Hay-fever, which was already known in former centuries, has found its definite explanation only in the last decades. It has not always, in medical circles, met with the attention to which it undoubtedly has a claim. As is known, it occurs every year at a definite period in spring with certain predisposed persons. The symptoms, known most likely to many of our readers, are graphically described by the author. The full outbreak of the illness is generally preceded by a preliminary stage of 1 or 2 weeks' duration. There is at first a slight irritation of the eyes with an itching in the inner corners, and sometimes sneezing fits are noticed during a prolonged stay in the open air. This stage is followed on a determined day by the full outbreak of the illness. The itching of the eyes increases so far as to become unbearable, the conjunctiva is highly reddened and œdematous, the eyes burn and run. The nose is affected in no lesser degree. The continuously increasing itching leads to long and violent sneezing fits; large quantities of a clear liquid are ejected, the mucous membranes of the nose are red and swollen, and it is impossible to breathe through the nostrils. In the further course, similar symptoms make themselves felt in the mouth; the mucous membranes start itching, and especially the palate is affected by a tickling sensation which may continue through the Eustachian tube right into the ears. In some cases, as the process goes on, serious breathing troubles occur which may even last through the night and often give the whole illness a rather threatening aspect. Finally, some of the patients feel a most troublesome itching of the skin. There is no fever, generally speaking, but the persons affected suffer to such an extent from depression and stupor, headache and irritability that they are compelled to give up their professional activity for days or even weeks.

The symptoms are not always of equal intensity, but occur in the form of more or less lasting fits of varying strength. These are specially disagreeable, above all in the open air, on hot, sunny days in windy weather, whereas the patients remain almost unmolested on rainy days. This state lasts 6 to 8 weeks on an average; the attacks lessen gradually in number and force, and disappear completely in the end. In northern

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[1]) *Deutsche med. Wochenschr.* **43** (1917), 110; *Chem. Zentralbl.* **1917**, I. 526. — [2]) *Zeitschr. f. ärztl. Fortbildung* **14** (1917), 292.

Germany, the hay-fever season begins in the second half of May; a little sooner, in southern Germany. The climax of the suffering coincides with the blooming of the grasses, especially of rye.

In the United States of North America, hay-fever occurs as "spring fever", but rather seldom in comparison with the so-called "fall fever", beginning early in September with similar symptoms and lasting for about 6 weeks. Hay-fever occurs chiefly in Germany, England and the United States, but is also found in other countries. Above all the higher civilized people, especially brain-workers, are attacked, and, it is said, men more frequently than women.

There has been no lack of attempts to explain the external cause of hay-fever; Elliotson was the first to ascribe it to pollen. Blackley gave this theory the first solid foundation by his investigations, as he proved that only during the hay-fever season there were considerable quantities of pollen in the air and that such pollen, obtained in a pure state, had the property of causing the typical symptoms on coming into contact with the mucous membranes of the nose or mouth of hay-fever patients. According to Blackley's view, any pollen was capable of causing hay-fever.

However, Blackley's observations, which for several years had been considered as authoritative, were not left uncontradicted. Under the influence of the first successes of bacteriology, one thought it necessary to consider hay-fever as an infectious illness. All sorts of bacteria were described as causing hay-fever. On the other hand, it was tried to prove that the exhalations of plants, aromatics and essential oils, such as are developed under the influence of sunshine during the first time of flowering, were responsible for the attacks of hay-fever.

It was reserved to Dunbar's investigations and to those of his collaborators Prausnitz, Kammann, and Liefmann, to make Blackley's old pollen theory valid again and to put it on a scientifically indisputable footing. Dunbar obtained pollen in a pure state by cutting grasses, or the ears thereof, shortly before flowering, placing them in water in a warm spot and making the pollen drop on glossy black paper by repeated rapping at the stalks.

Infinitesimal quantities of this grass pollen were then introduced into the conjunctival sack or into the nostrils of hay-fever patients by means of a sterile piece of cotton wool. With all of them, the typical symptoms of hay-fever appeared within a very short time, although nearly all the experiments were carried out in times when there is no hay-fever. Persons who do not suffer from hay-fever, however, were not at all influenced by this treatment. Furthermore, a small quantity of rye pollen was diffused in a cupboard where there were a hay-fever patient and a normal person; whereas the latter was not influenced in the slightest degree, the former felt the characteristic troubles already after a few minutes. The corresponding symptoms of irritation occurred with a hay-fever patient also when rye pollen was introduced into the anus, a proof that hay-fever is not due to a special sensitiveness of the trigeminus, as had formerly often been supposed.

So far, the experiments had only shown that the pollen of gramineæ may be looked upon as the cause of hay-fever, — the question was still open whether other kinds of pollen would act in a similar way and whether the quantity of pollen present in the air under normal conditions would suffice to bring about attacks of hay-fever. Dunbar and Lübbert's investigations, continued through several years and which comprised the examination of grasses and 106 other plants, showed that, in addition to the pollen of 33 gramineæ and cyperaceæ, only the pollen of honeysuckle *(Lonicera caprifolium)*, lily of the valley *(Convallaria majalis)*, *Polygonatum multiflorum*, common

evening-primrose *(Oenothera biennis)*, rape *(Brassica Napus)* and spinach *(Spinacia oleracea)*, as well as a number of compositæ, is efficacious with European hay-fever patients. The American autumnal catarrh (fall fever) is caused by the pollen of *Ambrosia* and *Solidago* species. American patients who only suffer from the spring catarrh, but not from the autumnal one, are only affected by grass pollen, whereas a third group of patients is sensitive towards the pollen of gramineæ as well as towards that of compositæ. Besides, also in Europe highly sensitive persons have been found, who are irritated by either kind of pollen. Finally, Dunbar succeeded in proving that privet pollen is the cause of the Chinese hay-fever and the pollen of an *Eragrostis* species, widely spread in South Africa, of that of the South African hay-fever.

According to Prausnitz and Liefmann, the pollen begins to increase in Hamburg already towards the end of May, reaches its maximum about middle of June, in marked coincidence with the intensity of hay-fever, and starts diminishing gradually in the second half of July, so that at the end of July or early in August only stray pollen are still found. With the aid of a special aeroscope, Liefmann was able to prove that, in the centre of the large town, 308 pollen were contained in one cubic metre of air on the 10th July and that in the neighbourhood of a rye field one takes in 2 or 3 pollen with every breath. As 40 to 50, with highly sensitive patients only 2 to 4, suffice to cause hay-fever, the figures given show that the pollen contained in the air are sufficient to cause the attacks.

The poisonous action of the pollen is due to a toxical albumen, which may be obtained from a pollen extract by precipitation with alcohol, or by dialysis. As per Kammann's investigations, the rye pollen protein prepared in this way is a thermostable poison, which remains unaltered on heating for one hour up to 60 or 70° and only on being boiled loses three-fourths of its original efficacity. It is not attacked by sulphuric acid, but potash lye, trypsin, and pepsin, combined with hydrochloric acid, destroy it to a great extent. The introduction of one drop of a 1:20 000 down to 1:40 000 solution of the poison, obtained by precipitation with alcohol, into one eye, or the nose, of a hay-fever patient will cause the characteristic symptoms within a few minutes, whereas normal people will remain completely unaffected. The subcutaneous injection of the poison was followed by violent attacks of hay-fever, according to Dunbar's observations. The method of preparing the poison has been further improved of late by Kammann, who succeeded in preparing a solution of it, which kept its efficacity unaltered for years and, even in a 300-millionfold dilution, brought about the typical symptoms in highly sensitive hay-fever patients.

The isolation of the rye pollen protein has enabled the physician to satisfy himself at all times as to whether a patient suffers from European or American hay-fever, or from some other illness (vasomotoric coryza, &c.). Instead of the pollen extract, not always available, and which must always be freshly prepared, he may use the so-called hay-fever diagnostic, which is quite harmless for otherwise healthy eyes and has become indispensable for differential diagnosis.

The improvement of the preparation of the pollen toxin proved useful also in that animals can be immunized with Kammann's preparation to a much higher degree than it was possible with the former protein. On treating them with subcutaneous or intravenous injections of pollen protein, certain bodies appear in the serum of these animals which are capable of neutralizing the poison. Already in his first publication, Dunbar was able to report that such an antiserum altered a solution of pollen toxin *in vitro* to such an extent as to make it absolutely ineffective towards hay-fever patients, and that furthermore the irritation caused through pollen toxin could be removed

again by subsequently applying the serum. Not all kinds of animals are fit for the preparation of the antitoxin. Especially horses, which distinguished themselves from the very outset by a certain sensitiveness towards poison, and certain rabbits supplied antisera of high value, whereas goats were generally indifferent to the pollen toxin and unfit to produce antitoxin. Appropriate horses, on subcutaneous injection of the extract of from 0,5 to 1 gram of rye pollen, partly showed tremendous swellings of from $^1/_2$ to $^3/_4$ m. diameter, high fever and serious general disturbances, partly less pronounced symptoms. On continuing the treatment with increased doses, no rise of sensitiveness was to be noticed, however; on the contrary, the animals got more and more accustomed to the poison and were finally able to stand 20 to 30 times the original dosis without any difficulty.

The serum from sufficiently immunized animals has poison-binding qualities. In opposition to Magnus and Friedenthal, Dunbar was unable to prove the presence of precipitating antisubstances, but binding antisubstances, which in some rabbit sera suppressed the hæmolysis even in pollen toxin diluted in the proportion of 1:50000.

A method worked out by Dunbar and Prausnitz permits of ascertaining the neutralizing effect of the serum on known quantities of toxin in the eye of the hay-fever patient. The Pollantin sold by us is 40- to 50-valent, on an average. The treatment of horses with Kammann's new protein enables one to arrive at a 60- to 80-, even at a 100-fold valency. The sera against the American fall fever are made and examined in a similar way.

As to the nature of the hay-fever toxin and the antitoxin, the opinions are divided even now.

Prausnitz succeeded in proving that, with increasing doses of rye-pollen toxin, the quantity of serum required for neutralization does not rise in constant proportion, but more quickly, so that the binding of toxin and antitoxin does not follow the law of the multiple proportions. At first, Prausnitz tried to explain this by a small avidity of the horse antiserum. On the other hand, Weichardt and Wolff-Eisner supposed that the hay-fever poison was not a true toxin, but an endotoxin, and the antiserum not an antitoxin, but a cytolytic amboceptor. Accordingly, the specific serum, in the presence of a suitable complement, would free the poison from the pollen and, in consequence, cause no relief of the hay-fever attack, but a change for the worse. Weichardt explains hay-fever as an illness caused by endotoxins, which are formed on pollen albumen being split up by cytolysins. Later on, he further developed this view in the sense of modern investigation of anaphylaxis, considering hay-fever to be a cellular epithelial anaphylaxis. Then, the hay-fever poison would be no preformed toxin, but would be formed by parenteral digestion of the primarily innocent pollen albumens in, or close to, the mucous cells of the conjunctiva and the respiratory tract of the hay-fever patient. The ineffectiveness of pollen toxin on the mucous membranes of healthy persons would then be explained by the absence of permeability for the noxious components, so that there was no possibility of parenteral cellular action.

Wolff-Eisner likewise takes hay-fever to be a supersensitiveness towards albumens and supposes that the serum of hay-fever patients contains substances which, through an endolytic process, set free the poisonous components of the pollen albumen. In accordance with Weichardt, he designates Pollantin, obtained from animals through injecting pollen, as a cytolytic serum which, in the presence of the required component, sets free the endotoxins, thus increasing the action of the poison. The uncontested successes of Pollantin were to be explained by the presence of bodies in the serum which hinder the reactions, the so-called "colloidal checking substances", *i. e.* suppress

the cytolysis of the pollen albumens like the antiferments. This action is said to appertain in a still higher degree to the normal serum (Graminol), owing to the absence of the cytolysins.

Finally, even Prausnitz declared of late that the anaphylactic hypothesis was the most satisfactory one, generally speaking. He sees the cause of the disposition for hay-fever in the presence of relatively small quantities of an amboceptor, specific for pollen albumen. Under its influence, the pollen albumen that reached the mucous membranes was changed from the complement into a poisonous intermediate product, from which finally, if the reduction continued, innoxious substances were formed. The success of the hay-fever serum was due to the fact that it created a sufficient excess of amboceptors, in consequence of which the resolution process was so quick that the poisonous intermediate product did not get any chance to act.

In opposition to said authors, Dunbar up to now holds that the pollen poison is a true toxin and Pollantin, in consequence, an antitoxin. For not only did Kammann succeed in proving that the *Ambrosia* poison was neutralized by his (Dunbar's) specific serum according to the law of the multiple proportions, but, in addition, Kammann and Gaehtgens ascertained in further investigations that the saturation of the rye pollen toxin by Pollantin strictly followed the same law. The experiments, even with 80 times the ordinary irritative dosis, resulted in that the toxin was bound completely by the corresponding (80 times) effective quantity of serum and thus showed that Pollantin must be looked upon not as a cytolytic, but an antitoxic, serum. According to Dunbar's investigations, further reasons speak against considering hay-fever as an anaphylactic illness. On the one hand, it is possible to produce an antitoxin against hay-fever, whereas it is characteristic for anaphylaxy that in no case any counterpoison for the anaphylatoxin has been noticed; on the other hand, Dunbar never succeeded in passively transferring anaphylaxy on guinea-pigs with the serum of hay-fever patients. Similar experiments with the serum of guinea-pigs, which had before been treated repeatedly with pollen albumen, yielded no definite results and can in no case be exploited in favour of the theory of anaphylaxy. Furthermore, there are no observations, so far, that the attacks of hay-fever were followed by a reduction of the sensitiveness towards pollen in the sense of anaphylaxy. In the contrary, the susceptibility of the patient rather seems to increase after each attack; at least, the sensitiveness remains unaltered for years and years. The view, that the occurrence of hay-fever was to be explained by that the sensitiveness was brought about through the contact with considerable quantities of pollen, cannot be maintained in every case, as Dunbar was frequently able to observe that persons who lived in Germany and had never been to America, therefore never had come into touch with the pollen of *Ambrosiaceæ* or *Solidaginaceæ*, got hay-fever nevertheless on the first contact with such pollen albumen. Above all, Dunbar succeeded in stating that a normal person, in spite of subcutaneous injections of rye pollen albumen in increasing doses continued for several weeks, did not become supersensitive and did not show the slightest symptoms of hay-fever at the critical period.

For all these reasons, Dunbar declines to designate hay-fever as a purely anaphylactic process. He thinks a complete explanation of the individual disposition for hay-fever is not possible so far, but as the established proof of the presence of antipollen albumens in the serum of the hay-fever patients points to the parenteral absorption of pollen substances by the human body, one would be entitled, at present, to look upon the symptoms of hay-fever as a defensive reaction against the parenteral introduction of genuine noxious pollen albumen through the abnormally permeable cutis and mucous

membranes. This state might be the consequence of a damaged vasomotoric apparatus, due to a violent influenza or other illnesses.

The prophylactic treatment of hay-fever may consist in spending the critical time in districts, where the air does not contain any pollen. In Germany, previous to the war, Heligoland was specially preferred by hay-fever patients as a place comparatively free from pollen. A further possibility of protecting the mucous membranes against pollen is to use goggles for the eyes and cotton wool filters for the nose, which keep away or filter the air laden with pollen and are said to have given good results. In addition, the continued use of calcium chloride is said to reduce the permeability of the vessels. Recently, Loew prepared a double salt under the name of "Kalzan" *(calcii et sodii lactas)*, which is supposed to be free from the drawbacks of calcium chloride, and the continued administration of which, even in large doses, is stated to cause no unfavourable by-effects. Dunbar did not gain the conviction that the resorption of the pollen albumen was prevented by calcium chloride. Hoffmann could not book either any incontestable success. Emmerich and Loew, however, report that taking 3 grams of calcium chloride crystals daily for several weeks before and during the hay-fever season might remove the most essential symptoms and that the use of the substance, if continued for years, might even free from the complaint altogether. Similar favourable reports are given by Kayser and Seiffert, the latter of whom, however, refrains from making a conclusive statement. Further observations must show whether the hopes set on the calcium treatment will be realized.

It is interesting to hear from Galisch, who had been suffering severely from hay-fever for 30 years, that he was free from any attacks in the two war summers 1915/1916, spent in the West as well as in the East. He thinks, his being cured may have something to do with having been hardened through exposure in the war. Such a possibility seems to be an exception, however, for Gaehtgens has had an opportunity of gathering from letters of soldiers at the front that even there they were not spared by their complaint.

According to the experiences of Dunbar, Lübbert, Prausnitz, Zarniko, and others, a considerable curing effect is decidedly due to Pollantin, as more than half of the hay-fever patients succeed in keeping themselves almost entirely free from any attacks by properly using the remedy. Although it is easily handled, it is often applied in the wrong way, a fact which accounts, beyond any doubt, for a good many failures, for which Pollantin is blamed. One of the chief rules, against which many patients sin, is that only *small* quantities of the preparation ought to be conveyed to the mucous membranes of the eyes, the nose and, if necessary, the mouth, *before* any strong irritation has been noticed. It is wrong to take large doses, as they mechanically irritate the mucous membranes and may cause supersensitiveness. It is wrong too to use the remedy after the symptoms have fully developed, as then the mucous membranes are only irritated further and a resorption through the dropsically swollen mucous membranes does not take place any more. A further cause of Pollantin refusing to act occasionally may be a simultaneous supersensitiveness towards horse serum, which is acquired by some patients in the course of the treatment. The introduction of one drop of immunized or ordinary horse serum, during or out of the hay-fever season, brings about in such persons symptoms of irritation similar to those of hay-fever. But there is help for such persons in two ways. Either they may be recommended to use the diluted Pollantin "R" in very small quantities and, if possible, only once daily, or, in some cases, when the patients had become anaphylactic towards horse serum, the application of an immunized serum of another kind of animals, *e. g.* rabbits,

led to the desired effect. It is a pity, however, that in such cases an anaphylaxy also towards rabbit serum seems to develop within a rather short time, as Dunbar has been able to observe.

It is recommendable, therefore, to introduce infinitesimal quantities of Pollantin into the eyes and nostrils already in the morning before getting up and to repeat the process as soon as the slightest irritation is noticed in the course of the day. Needless to say, it is advisable to combine the treatment with a rational prophylaxis, *i. e.* to keep the windows shut in the bed-room during the time when the grasses flower and then to avoid any unnecessary stay in the open air, especially in dry weather, &c. As a rule, by following these hints, patients may keep themselves free from hay-fever or, at least, reduce the symptoms to a bearable degree. As per the experiences of Dunbar and Albrecht, the due use of Pollantin in many cases not only reduced the sensibility towards pollen, but cured the illness completely for good.

It cannot be denied that the hay-fever patients find it often inconvenient to use the various remedies and that, above all, the serum only affords protection for a limited space of time. Therefore it is easily understood why attempts have been made to arrive at a lasting immunity through active immunization, a question, the solution of which seemed all the more promising as observation teaches that, in many persons, the disposition for hay-fever decreases with age.

The first experiments in this direction were carried out by Noon and Freeman, who subcutaneously treated a rather considerable number of hay-fever patients, in some cases for months, with increasing doses of an extract of the pollen of *Phleum pratense* (Timothy grass). Objectively, it was possible to state regularly a considerable decrease of the sensitiveness towards pollen, in some cases down to $^1/_{100}$ of the original sensitivity. The clinical successes were generally rather satisfactory, as they were complete in three cases, out of 18 treated by Freeman, whereas there was a more or less pronounced improvement in 13 and only two gave no result at all. Dunbar gathered similar experience in treating ten hay-fever patients. His clinical successes could be called fairly satisfactory, generally speaking, especially when taking into consideration the short period of treatment, the majority of the patients having had to complain only occasionally of hay-fever symptoms, and then mostly of slight ones, during the critical period.

Lovell, Sormani and Wolff-Eisner likewise report on good results with active immunization of hay-fever patients. Besides, Ellern treated 13 patients with Wright's pollen vaccine; however, he does not consider as a proof of the value of the process the considerable improvement, declared by nearly all the patients, as 16 out of 20 other patients, who had *not* been treated, stated to have noticed an improvement of their complaint in the same summer.

Finally, Gaehtgens gives a short report on experience gathered in 1913, 1914 and 1916, when continuing Dunbars investigations as to the value of active immunization. The treatment was started from 8 to 12 weeks before the hay-fever season and concluded towards the middle or end of June, unless exterior reasons caused a premature interruption. The injections were carried out subcutaneously, and at intervals of from 5 to 6 days, with Kammann's new toxin preparation from rye pollen. The initial dose was, as a rule, one fifth of what had been ascertained to be the limit of sensitiveness by means of the ophthalmo-reaction, the quantity being doubled with each consecutive injection, unless a stronger reaction after the last injection made it advisable to give the same dose once more. In this way, some patients were able to have from 16 to 18 injections during one treatment. On an average, the initial dose was

1 c.c. of the dilution of 1 in from 5 to 10 millions, the final dose in most from 1:20000 to 1:2000, from which it results that it had generally been possible to raise the dose 1200 fold in the course of an uninterrupted treatment. The sensitiveness of the eyes sank at the same time to from one tenth to one twentieth of what it had been, so that it can only serve as a criterion for the initial dose, whereas after repeated injections the clinical reaction of the previous injection only can serve as a guide for the determination of the fresh dose. The injections were sustained well, on a whole. The local reaction after the injection generally kept within moderate limits, was gone as a rule after 1 to 3 days, and lessened in intensity from injection to injection. General symptoms were noticed only once with a highly sensitive lady patient, who, half an hour after the injection, got a violent itching of the skin all over the body, a rash on neck and arms, swelling of the face, and experienced difficulty in breathing; however, these symptoms had gone the next morning. At the same time, the limit of sensitiveness had risen again from 1:500000 to 1:1000000.

In order to judge of the clinical success, two of the eight cases treated must be left out of consideration, as the treatment had to be interrupted after two and eight injections, respectively. Three of the remaining ones called special attention, owing to their having been treated already by Dunbar in 1912, two with good, and the third with less success. One of these three persons was treated again in 1913 and 1914; the second in 1913, and the third in 1914. With the first lady patient, who was highly sensitive and anaphylactic towards horse and rabbit sera and who had undergone the treatment three times, the specific therapeutics caused no improvement worth mentioning, so that this case had to be booked as a failure. The results obtained with the other five persons, however, were rather satisfactory. One of the cases, immunized for the second time in 1913, was specially successful, as the patient was almost free of his old complaint without any treatment in summer 1914.

Summarily speaking, the value of active immunization causes in many patients an unmistakable alleviation of the attacks of hay-fever and, in some cases, seems to lead to an almost complete cure of it. However, according to the experience gathered up to now, the hope to attain a lasting immunity through the injection, has only been partly realized. One ought, therefore, to beware of overrating the treatment, which, by the way, highly taxes the patience of the patients. On the other hand, however, the observations made entitle us to recommend, in appropriate cases, the treatment of the complaint through active immunization.

Thymol. On page 78 of our last October *Report*, we discussed the *Bulletin* N° 372 of the U. S. Dep. Agric. by S. C. Hood according to an abstract in the *American Journal of Pharmacy*. As we now have the original paper at our disposal, we seize the opportunity to supplement our first communication.

Monarda punctata often only developes very few leaves which reduces the oil yield to a great extent. By means of selection, Hood succeeded in obtaining a larger and lighter coloured plant which produces more leaves and also grows to a greater height. It contains about 0.42 per cent. of oil with 74 per cent. of phenols, whereas the original plants in the first year supplied 0.17 and in the second 0.24 per cent. with a phenol content of 64 per cent.

In the South-eastern states of North America the seeds of *Monarda punctata* mature in August and September. In the extreme South-eastern states, where no severe frost nor heavy snowfalls are to be feared during winter, sowing can be done already in November. The most suitable soil for this cultivation is light, calcareous and sandy

in quality. It is advisable to place the plants in rows 3 feet apart and the latter must be $1^1/_2$ to 2 feet apart. A mixture of ammonium sulphate, acid phosphate and potassium sulphate is recommended as manure. It is imperative to distil the herbs in a fresh state, immediately after having been cut, because during the drying process not only oil is lost, but the leaves also fall off easily which causes a further loss in oil. The best time for gathering the plants is when they begin to develop their blossoms, but before they are in full bloom. This statement is illustrated by the following table:—

Plants just beginning to send up flower stalks	0.34 per cent. of oil with	72 per cent. of phenols,
Budded stage	0.30 „ „ „ „ „	76 „ „ „ „ „
Full flower	0.24 „ „ „ „ „	74 „ „ „ „ „
Flowers fallen	0.18 „ „ „ „ „	74 „ „ „ „ .

For distillation purposes, Hood recommends the same apparatus used for the production of peppermint and spearmint oil. It is even possible to obtain a further supply of 0.114 per cent. of oil with 95 per cent. phenols from the distillation water. It is occasionally advisable to cut the herbs, but this has no effect on shortening the time necessary for distillation.

Hood recommends fractional distillation for isolating the thymol from the oil. The phenols of the oil consist almost entirely of thymol and contain hardly any carvacrol; the non-phenols consist in the main of cymene. Thymol can be separated easily in a pure state from the oil by fractionating it; Hood obtained from an oil that contained 72 per cent. of phenols by distillation 64.3 per cent. of thymol. He does not refer at all to the melting point of thymol. The distillation residue consisted of a thick, tarry mass which has hitherto found no application.

A good mouth wash must prevent the growth of the bacterium *Leptothrix buccalis* which is the cause of the formation of tartar and probably also of pyorrhœa. For this purpose, the teeth must be treated as often as possible with a mouth wash by the aid of a tooth brush which purpose is best served according to H. P. Goodrich[1]) concentrated watery thymol solution. It has a pleasant taste, acts as a strong antiseptic and in a less irritating manner than carbolic acid, neither does it act as an acid nor does it possess the corrosive effect of the above-mentioned acid. Besides, it is quite harmless if swallowed by mistake. The following compounds are less effective according to Goodrich:— boric acid, emetin hydrochloride, iodine, hypochlorite solution, peroxide of hydrogen, potassium permanganate, and flavine.

While studying substances which act hæmolytically, especially extracts of plants, it has often been the practice to to treat the solutions with thymol. This is not admissible, as P. Laviałle and A. Aubry[2]) have found, and has in consequence often led to false statements because thymol *itself* acts as a strong hæmolytic. The hæmolytic action of thymol depends on its concentration, saturated solutions having a stronger action than weaker ones, besides which the effect is dependant an the temperature and on the presence of various acids.

Vanillin. On page 80 of our last October *Report* we discussed a publication by Constantin and Bois on the three varieties of *Vanilla planifolia* which are cultivated on Tahiti. This paper has now been supplemented by a further publication of the same authors[3]).

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[1]) *British medical Journ.* **1917**, I. 473; *Chemist and Druggist* **89** (1917), 376. — [2]) *Bull. Sciences pharmacol.* **23** (1916), 193, 266. — [3]) *Compt. rend.* **163** (1916), 466.

In a treatise published in 1829, Schiede[1]) refers to four species of vanilla found in Mexico, namely:— *Vanilla sativa*, *V. sylvestris*, *V. Pompona*[2]) and *V. inodora*. The latter has been confounded with the cultivated variety *V. sativa* and called by Swartz *Vanilla aromatica*, although the fruit has no smell whatever.

According to Schiede, *Vanilla sativa* ("the Baynilla mansa" of the Spanish-Mexicans) is the best sort of vanilla and is cultivated in Mexico, Papantla, Misantla, and Colipa. From more recent information it appears that it is specially grown in the state of Veracruz and that it exists in a state of half-culture in the surrounding forests. The aborigines cultivate the plants which are most suitable for that purpose in the woods and even in the neighbourhood of their dwellings. They treat the pods with particular care in reference to the formation of aroma, whereby they show great skill, as they can depend on the experience of past generations. They are especially experienced in all matters concerning fermentation.

Vanilla sylvestris ("Baynilla cimarrona") is cultivated in southern Mexico, at Papantla, Nantla, and Colipa.

In reference to the vanilla varieties found on Tahiti, the authors have been able to study three forms, the Tahiti vanilla, the Tiarei vanilla and the Haapape vanilla which appears to be related to *Vanilla sylvestris*. According to their publications the nomenclature of the vanilla varities would be the following:—

Vanilla planifolia, Andrews var. *sativa* = Mexico vanilla.
" " var. *sylvestris*.
" " subvar. *angusta*, Constantin et Bois = Tahiti v.
" " " " " " " = Tiarei v.
" " " " " " " = Haapape v.

Vanilla has been cultivated for the last 50 or 60 years on Tahiti. Originally only two varieties were known there, the Mexico vanilla and the Tahiti vanilla. Although the Tahiti vanilla produces smaller pods and with a less finer aroma than the Mexico vanilla, it is much more appreciated by the planters because the pods on becoming mature do not open and because accordingly one can let them get black while remaining on the plants. One often leaves the pods on a heap for 2 or 3 weeks after gathering them before they are dried in the sun. Considering the state of affairs on Tahiti, where it is difficult to obtain any manual labour and where the aborigines are exceedingly indolent, this variety is most suitable. Mexico vanilla must be treated with much greater care.

The two varieties Tiarei and Haapape appeared quite suddenly on the island. The former was observed for the first time about 10 years ago in the Tiarei district. It only blossoms rarely and has to be treated with great care. The Haapape variety was observed for the first time about 2 years ago in a neighbouring district; it bears blossoms in quantities twice a year.

The commercial value of the three varieties is the same. The pods have the same aroma, reminding of heliotrope like the Tahiti vanilla.

Several methods are known for determining vanillin which are mostly based on its conversion into compounds which are difficultly soluble. We recall the methods of Hanuš[3]), founded on the formation of very insoluble compounds of vanillin and β-naphthylhydrazine or with *p*-bromphenylhydrazine and those of Feinberg[4]) (conversion

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[1]) "Botanische Berichte aus Mexico", *Linnæa* **4** (1829), 573. — [2]) The mother-plant of the "vanillons". — [3]) Comp. Gildemeister and Hoffmann, *The Volatile Oils*, 2nd ed., vol. I, p. 588. — [4]) *8th International Congress of Applied Chemistry*, Washington and New York 1912, vol. I, p. 187. — *Americ. chem. Journ.* **49** (1913), 87; *Report* April **1913**, 124.

of vanillin into its *p*-nitro-phenylhydrazone). Colorimetric methods have also been elaborated, for instance Fellenberg[1]) has suggested a process, which depends on the colour-reaction which vanillin undergoes with conc. sulphuric acid and *iso*butyl alcohol. A. W. Dox and G. P. Plaisance[2]) have elaborated a new method for testing the alcoholic vanilla extracts which find so much favour in America; they have found, namely, that thiobarbituric acid forms with vanillin in the presence of 12 per cent. muriatic acid an only slightly soluble vermilion-coloured precipitate of 3-methoxy-4-hydroxybenzalmalonyl-thiourea. This method is not suitable for the valuation of artificial extracts coloured by caramel, as the latter contains furfurol which also reacts with thiobarbituric acid. Accordingly, the vanilla extracts must be tested for the presence of caramel, for which purpose one adds phloroglucinol to the solution, previously clarified by acetate of lead, and after having removed the excess of lead as chloride of lead. A brown precipitate is formed in cases in which caramel is present; when absent, however, the vanillin only undergoes a light-pink colouring or forms a small quantity of a pink-coloured precipitate.

The determination is executed in the following manner:— 25 ccm. of the alcoholic vanilla extract are freed from alcohol in the customary manner and filled into a measuring flask of 50 ccm. volume ("Sugar flask"), which is then filled up to the graduation mark with lead acetate solution. The flask is then left alone at 37° for some hours an its contents are then poured on to a dry filter. 40 ccm. of the filtrate which must be straw-coloured (absence of caramel) are then poured into a second 50 ccm.-flask and filled up to the mark with concentrated muriatic acid; the acid content must then be about 12 per cent. In order to remove the lead chloride, after a few minutes one filters the liquid through a dry filter, treats 40 ccm. of the filtrate with a solution of thiobarbituric acid in 12 per cent. hydrochloric acid, lets it stand over night, filters through a Gooch crucible, washes with 50 ccm. of 12 per cent. muriatic acid, finally with 20 ccm. of water and dries at 98°; one can then easily calculate the content of the extract in the shape of vanillin from the weight of the precipitate. Clarification with the aid of lead acetate is imperative, as otherwise the results are too high.

C. Estes[3]) has described another method for the determinatidn or at least for the qualitative detection of vanillin. If one heats vanillin with mercuric nitrate, a violet colouring is formed whose intensity corresponds to the quantity of vanillin present. One treats 5 ccm. of vanilla-extracts, in a 50 ccm.-flask, with 6 ccm. of water and 1.5 ccm. of the reagent (see below); after leaving the flask for 20 minutes in boiling water one cools it down rapidly, the contents are diluted to 50 ccm. and the colouring formed compared with that produced by a known quantity of vanillin under the same conditions. Non-alcoholic extracts are treated just in the same manner, but in such cases one only requires 1 ccm. of the reagent. For the purpose of preparing the latter, one dissolves mercury in the double quantity of nitric acid (specific gravity 1.42) and dilutes the solution with 25 times as much of water.

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[1]) *Mitt. Lebensmittelunters. u. Hyg.* **6** (1915), 267; *Report* October **1916**, 92. — [2]) *Americ. Journ Pharm.* 88 (1916), 481. — [3]) *Journ. ind. eng. Chemistry* **9** (1917), 142; *Journ. Soc. chem. Industry* **36** (1917), 352.

Notes on Scientific Research in the Domain of the Terpenes and Essential Oils.

General.

Homage to Wallach on his 70th birthday. — A. Binz[1]) wrote an article for Wallach's 70th birthday, in which he sketched in short lines the merit of this scientist as far as our special branch of science is concerned and in which he points out specially, besides, Wallach's educational talent. He concluded it with the words: "If we consider what an amount of self-denial is required for a great scientist, such as Wallach, to sacrifice his time, as he is wont to do, with unvarying conscientiousness and enthusiasm to young people, we understand what Wallach signifies to us. He belongs to those who may be called educators of the nation and to whom we are indebted for having become strong enough to hold out in the struggle".

From other quarters too, homage has been paid to Wallach on his 70th birthday: A. Hesse[2]) gives a short review of Wallach's career and the origin of his life-work, the study of the chemistry of the terpenes and volatile oils. He mentions, *e. g.*, a passage out of Wallach's speech in which he thanks his pupils on the occasion of the celebration on August 4th, 1909. In this speech, he described in a few words the genesis of his work: in a cupboard in Kekulé's private laboratory, there had been kept unopened, for sixteen years, some bottles of essential oils, which Kekulé had ordered occasionally for investigation, but without making any use of them. Wallach, as a professor of pharmaceutical chemistry, had experienced already the lack of knowledge concerning the essential oils, which are so important for dispensing chemists. Kekulé gave the permission asked for to investigate the mysterious contents of the bottles with an ironical smile, said to have been peculiar to him whenever he thought anybody to be on the wrong track, and with the words: "If you think you can make anything out of them!"

We owe to Wallach, and to him alone, not only the successful scientific study of the domain of the terpenes and essential oils — for the numerous investigations of other scientists have become possible only through his work — but, besides, he laid the foundation for the development of a whole branch of industry, with methods formerly based on purely empirical experience, into an industry working on scientific principles.

The perfume industry in France. — Even up to now, like fashion, French articles of perfumery enjoy a renown, such as it has been impossible so far for any other producer to acquire for his products. There are experts who ascribe to nothing else but a prejudice of the public at large the repute of French perfumes and cosmetics. According to R. Löffl[3]), this is not quite founded on fact.

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[1]) *Deutsche Parf.-Ztg.* **3** (1917), 76. — [2]) *Zeitschr. f. angew. Chem.* **30**, I. (1917), 81. — [3]) *Seifensiederztg.* **44** (1917), 150, 173, 195.

The superiority of the French products is not due to the raw material. As far as the intermediate products of perfumery are concerned, such as are obtained from imported drugs, the Saxon products are at least equivalent in quality to those from Marseilles, Nice and Grasse, and superior in cheapness, *i. e.* out of two aniseed, sandalwood, caraway, or fennel oils, &c., equal in quality, that of German make is nearly always cheaper, as apparatus and working methods are better, more exact and cheaper in Germany, and the work also being done more quickly there. In this respect, there is nothing to be improved. The conditions are similar with oils imported in a crude state and purified, or otherwise improved, in the country. In these two sections, Germany has, besides, a scientific merit, for although German investigation in this domain began considerably later, it surpassed all the others in results. The whole world has acknowledged the German discoveries how to prepare synthetically new bodies with the aid of the exact analysis of the components of the oils and the determination of the physical and physico-chemical (among these especially the stereochemical) properties and constants, discoveries connected with the names of Wallach, Baeyer, Tiemann, Semmler, Gildemeister, and Rupe. There is no wish to belittle or to obscure the work of English, American and French scientists, but he who impartially studies the relative publications must say that the lion's share in the development of the chemistry of the essential oils and aromatics belongs to the Germans.

As regards the manufacture of the common oils, such as those of wormwood, valerian, tarragon, melissa[1]) and especially lavender and thyme, Germany does not grow them to any considerable extent, nor own any regions where they grow wild, so as to afford sufficient raw material. Knowing the present producing countries or districts, Löffl considers as unsuitable the recommendation to start in Germany growing these plants, or peppermint and other medicinal herbs, as likewise the cultivation of most of the plants producing oil fruits or seeds, *e. g.* sunflowers, for which so much propaganda is made just now. According to his view, lavender, thyme, wormwood[2]), &c., will never thrive with us in equal quality as in Spain, the South of France and round Beyroot. The economic reason for which Germany is not fit for this kind of cultivation is that the ground is too expensive and the work of gathering likewise.

The treatment of the commoner kinds of raw material, such as lavender and thyme, is better with us in many cases, and French manufacturers have often indirectly been induced to employ better and more rational methods by German firms who, like Schimmel & Co., settled down in the producing districts.

In this respect, there is nothing to be altered and little to be improved in the German industry. Where we have no raw material in the country, the production is not to be forced, the only way being to erect small special branches. Especially the countries who are friendly disposed towards us, such as Bulgaria and the producing districts in Asia Minor, are to be thought of in this way, and Syria, *e. g.*, where Lautier had a remunerative branch at Beyroot, is a region worthy of note, where plantations might be started on a large scale. In Asia Minor too, districts suitable for a similar purpose might be found, and it is there that we ought to send our botanists and chemists after the war. The necessary work of clearance and cultivation is comparatively easy there, all the more so as a good many of these plants require but little care, if soil and climate are favourable.

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[1]) The oil of melissa of commerce is no pure distillate, but either lemon or citronella oil distilled over melissa herb, if not simply a fractionated oil of citronella. The yield from the herb of *Melissa officinalis* L. is so small that the price of the genuine oil would be prohibitive. See Gildemeister und Hoffmann, "*Die ätherischen Öle*", 2nd ed., vol. III, p. 501. — [2]) This is not the case as far as wormwood is concerned.

But the production of raw material is not everything, especially in the manufacture of aromatics. Here, according to Löffl's view, manufacture and technics must be altered, if it has not been done yet, as in some isolated cases. With most of the fine aromatics, the extraction ought to be carried out in the cold with the aid of fat and alcohol, this being the first condition for obtaining really fine, first-rate perfumes equal to the French. After having adopted this method of preparing the intermediate products, we shall have facilitated considerably the perfumer's work.

Furthermore, the qualities and ability of the salesmen cannot be overrated. If one means to manufacture first-rate goods, one must sell them in a first-rate manner, a proceeding which Löffl explains by giving several instances.

The production of volatile oils in the Orient. — So much interest being shown just now to everything connected with the Orient, it seems appropriate to tell our readers something about the production of essential oils in Syria. According to A. Ruppin[1]), the volatile oil of the flowers of *Acacia farnesiana* has been often prepared in Syria, but with varying success. Factories, in which the odour of said plant, and of others, was concentrated in fats, were founded by a French company on the Carmel, the Jewish colonies of Sichron Jacob and Petach-Tikwah and in Jaffa, but they were abandoned a few years ago. A larger factory (likewise with French capital) near Beyroot carries on the extraction of floral odours and their absorption by means of plates of spermaceti, as well as the preparation of essential oils; it works with success. The fats imbued with odour and the crude essences are sent to Grasse, and there made into perfumes.

In addition to mimosa oil, the following were prepared: —

Oil of thyme[2]), from wild thyme, in various small distilleries in the Judaic mountains, *e. g.* the Jewish colony of Artuf between Jaffa and Jerusalem, in a primitive way. The oil finds a fairly good sale in Germany[3]).

Oil of geranium, the manufacture of which was introduced by Baron Rothschild. It is also obtained by a simple distilling process in the Jewish colonies of Rischon le Zion and Petach-Tikwah, where some colonists grow the geranium plant for the purpose.

Oil of laurel berries[4]) is prepared from the fruits of the laurel tree near Antiochia and in the South of the vilayet of Aleppo. A trial on a small scale has recently been made in the Jewish colony of Rosch-Pinah, near Safed. The oil is used in the manufacture of scented soaps in the soap works of Aleppo, Antiochia, and Tripoli.

Orange-flower water is made in Tripoli and sold to Egypt or to the French perfumery works at Beyroot.

Oil of aniseed is produceed in Galilee from the wild anise by means of small transportable stills. The aniseed cultivated near Damascus is partly exported to Europe and partly used in the manufacture of arrack (?).

A Jewish perfume factory in Jaffa uses the native oils for the production of Eau de Cologne and other scents; a similar factory in Tripoli employs both native and European oils, but the main part of the volatile oils distilled is exported to France and Germany.

In another place[5]), Ruppin gives some information concerning the importance of the exports of oranges, lemons, &c., from Syria.

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[1]) *Seifensieder-Ztg.* **44** (1917), 74. — [2]) Comp. *Report* October **1911**, 88. — [3]) Presumably this is a confusion with origan oil, which likewise contains phenoles. — [4]) *Reports* April **1909**, 65; October **1911**, 60. — [5]) *Beihefte zum Tropenpflanzer* **16** (1916), 421.

According to the Turkish official statistics, the exports to foreign countries were as follows: —

	kilos	value in piasters
via ports controlled by the Alexandrette customs-office	2 551 625	2 227 516
„ Beyroot	83 460	93 571
„ other Syrian ports	28 055 310	23 280 704
	30 690 395	25 601 791

As per the English Consular Reports, the exports to foreign countries and to Turkey amounted to: —

number of cases:

	1909	1910	1911	1912	1913
from Jaffa	744 463	853 767	869 850	1 418 000	1 608 570
„ Tripoli	134 000	218 000	135 000	140 000	—
„ Saida	188 000	125 000	70 000	79 600	—
„ Beyroot	3 200	1 200	4 800	1 860	—

value in francs:

from Jaffa	4 650 000	5 875 000	5 440 000	7 100 000	7 450 000
„ Tripoli	600 000	900 000	600 000	600 000	—
„ Saida	800 000	500 000	300 000	300 000	—
„ Beyroot	10 000	5 000	15 000	6 000	—

The Jaffa oranges, which can stand a longer journey, are mostly sent to Liverpool, in cases containing 144 and weighing about 35 kilos. During the shipping season (November to March), English ships go to Jaffa specially for the transport of oranges. It takes them 17 to 20 days to get from there to Liverpool. The freight from Jaffa to Liverpool generally comes up to fr. 1.50 per case. The other outlays of the producers, *i. e.* for picking and packing the fruit, &c., amount to 2 or 2½ fr. per case. The average price fetched by the oranges in Liverpool being 7 to 8 fr. per box, there is a net profit of 3 to 4 fr. for the producer. Sometimes, higher prices are obtained, but it may likewise happen that, the Liverpool market being overstocked, the oranges have to be sold at considerably lower rates, or that they arrive in bad condition and not even cover the outlays. This risk is the most disagreeable drawback of the otherwise very remunerative orange growing. In addition to Liverpool, Jaffa oranges are exported to Egypt (larger cases, inferior packing and second class fruit), Smyrna, Constantinople and Odessa. Attempts have even been made to start an export to Australia and the U. S. A., however, no definite judgment can be passed as yet regarding the success.

The exports from Jaffa during the season 1913/14 were as follows: —

to Liverpool	887 481 cases
„ Manchester	400 „
„ London	4 626 „
„ other English ports	3 361 „
„ Marseilles	3 412 „
„ Hamburg	9 487 „
„ Trieste	58 492 „
„ Odessa	148 409 „
„ Egypt, the Red Sea and Australia	149 846 „
„ Turkish ports	268 942 „
„ Roumania and Bulgaria	19 405 „
total:	1 553 861 cases

The largest part of the Saida oranges remains in the country, for they cannot stand a long journey, owing to their being thin-skinned. In consequence, only small quantities go to Constantinople, Odessa, Roumania and Liverpool, but fairly large quantities of lemons. Tripoli exports oranges and lemons, about one half to Odessa, a quarter to Constantinople and the remaining quarter to Salonica, Alexandria and Liverpool. The price in Tripoli was of late years about 8 to 10 fr. per case of 300 lemons and 4 to 5 fr. per box of 250 oranges.

The Jaffa oranges are partly exported by dealers, who buy them before they are ripe, either at a lump sum for the whole crop of a plantation, or at a fixed price per case. As the oranges ripen in November, but may remain on the trees up to March or April without damage, the dealer has plenty of time for picking them. The Jewish orange growers in the neighbourhood of Jaffa have formed two unions for the sale of their produce, and the German ones have followed their example; but so far it has been tried in vain to bring the Arabian owners of orange trees to act in a similar way.

The union decides when and where the oranges of its members have to be sent. Every member must put a certain mark on his cases, so that it is possible to give each a special account of sales, thus rewarding them for the good condition of the produce or making them responsible for bad quality.

The production of volatile oils in the U. S. A. — The industrial census[1]) of the U. S. A. for 1914 contains numerous figures regarding the essential oil industry, it being taken into consideration, however, that they are not definite as yet[2]). Information was given by 108 works, whose production represented a value of $ 2565361, comprising essential oils = $ 1289482, 917690 gallons of witch hazel extract[3]) = $ 575938, and other products = $ 699941. The census for 1909 mentioned 74 works with a production worth $ 1773304, of which $ 1111805 represented essential oils, $ 419793 witch hazel extract (691823 gallons), and $ 241706 the other products. In 1914, the total production amounted to $ 792057 or 44.7 per cent. more than in 1909. These products do not include synthetic or artificial oils, a considerable quantity of which is manufactured, especially artificial oil of wintergreen. For the year 1914 the production of two, and for 1909 that of six, works is mentioned, which in the main make other articles.

Inclusive of by-products and of the oils manufactured for others, the total production in 1914 was as follows: 363991 lbs. of peppermint oil to the value of $ 601617, 94209 lbs. of spearmint oil = $ 238074, 41178 lbs. of sweet birch oil = $ 67691, 6000 lbs. of wintergreen oil = $ 24538, 4702 lbs. of wormwood oil = $ 9040, and camphor, cedar wood, clove, lemon, parsley, patchouly, pennyroyal, sandal wood, sassafras, tansy oils, &c. to the value of $ 348522. In 1909, the output of essential oils of all the factories amounted to: 305781 lbs. of peppermint oil = $ 519079, 33400 lbs. of spearmint oil = $ 83283, 67053 lbs. of sweet birch oil = $ 102045, 22281 lbs. of wintergreen oil = $ 68983, 1989 (4989?) lbs. of wormwood oil = $ 9514, and other essential oils to the value of $ 328901.

The increase of the production of essential oils was 16 per cent. in five years. The increase of the production of peppermint oil was 19 per cent. in quantity and 15.9 per cent. in value, that of the production of spearmint oil, 182.1 per cent. in quantity and 185.9 per cent. in value. There was a decrease of 38.6 per cent. in quantity and 33.7 per cent. in value in the production of sweet birch oil and of 73.1 per cent.

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[1]) Comp. *Report* April **1904**, 106. — [2]) *Americ. Perfumer* **11** (1916), 95. — [3]) From *Hamamelis virginiana*, L., a shrub indigenous in the U. S. A., the bark and leaves of which are used for preparing an extract much esteemed in America. It is employed against various complaints, but its efficacity seems to be rather problematical.

in quantity and 64.4 per cent. in value in the manufacture of wintergreen oil. The output of wormwood oil and other volatile oils showed an increase in value of 5.7 per cent. The production of witch hazel extract rose by 32.6 per cent. in quantity and 37.2 per cent. in value.

Of the 108 works to which the 1914 report refers, 53 were in Michigan, 29 in Indiana, 7 in Connecticut, 6 in New York, 3 in New Hampshire, 3 in New Jersey, 2 in in Pennsylvania and 1 each in California, Kentucky, Massachusetts, Tennessee and Wisconsin.

Hereafter, we put before our readers once again the statistical data for 1914 and 1909 in the form of tables: —

	1914	1909	increase or decrease
number of factories	108	74	+ 45.9 per cent.
total value of the products	2565361 dollars	1773304 dollars	+ 44.7 „ „
values and quantities of the several oils:			
peppermint oil	363991 lbs.	305781 lbs.	+ 19.0 „ „
	601617 dollars	519079 dollars	+ 15.9 „ „
spearmint oil	94209 lbs.	33400 lbs.	+ 182.1 „ „
	238074 dollars	83283 dollars	+ 185.9 „ „
sweet birch oil	41178 lbs.	67053 lbs.	— 38.6 „ „
	67691 dollars	102045 dollars	— 33.7 „ „
wintergreen oil	6000 lbs.	22281 lbs.	— 73.1 „ „
	24538 dollars	68983 dollars	— 64.4 „ „
wormwood oil	4702 lbs. 9040 dollars	338415 dollars	+ 5.7 „ „
other essential oils	348522 dollars	328901 dollars	—
witch hazel extract	917690 gallons	691823 gallons	+ 32.6 „ „
	575938 dollars	419793 dollars	+ 37.2 „ „
all other products	699941 dollars	241706 dollars	—

The figures published by the Census Bureau seem to be open to criticism in several respects. The periodical, *e. g.*, from which we copy the census report, points out that, without any doubt, the production of wintergreen (gaultheria) oil has increased since the "pure food law" came into force, but it is impossible to indicate with any claim to an even only approximate accuracy how great this increase may be. The quantities manufactured by the various producers are small and represent but a small fraction of what is consumed of its substitutes, *i. e.* sweet birch oil and artificial wintergreen oil (methyl salicylate).

The indication regarding the decrease of the production of birch bark oil is said to be almost correct, the cause of it being, it is supposed, the exhaustion of the raw material.

The figures published by the Census Bureau show for 1914 an increase of 19 per cent. over 1909 in the production of peppermint oil, whereas it results from private statistics that the increase is far more considerable, 1914 having been a "bumper" year with a crop of more than 600000 lbs. The annual production is now estimated at about 400000 lbs., against about half this quantity twenty years ago.

The enormous increase in the production of spearmint oil will have struck our readers. We have repeatedly discussed the subject in previous *Reports*[1]). It is im-

[1]) *Reports* April **1908**, 99; April **1911**, 106; April **1912**, 117; October **1912**, 103; April **1913**, 96; October **1913**, 99; October **1915**, 36.

possible to predict whether the demand will continue; the latest news seem to indicate that there is a decrease in the consumption.

The cultivation of aromatic plants. — In a treatise about the cultivation of important medicinal plants, B. Pater[1]) says something about angelica. Only fresh seeds germinate, and if possible the seeds of the same year ought to be taken for sowing in autumn. Pater's self-grown seeds germinated, it is true, when sown in the following spring, but the results were better with autumn sowing.

Elecampane thrives better after autumn sowing, although spring sowing may be successful as well; sometimes, however, this fails altogether, owing to drought. Coriander, valerian, white and black mustard, hyssop and rue prosper also when sown in spring. Mustard, which germinates very quickly, must even been sown in spring.

Cultivation of aromatic plants in the Caucasus. — A good many years ago already, it was tried to rear plants in the Caucasus yielding essential oils, and it is said that in the beginning of this century an exellent otto of roses was obtained in Kachetia (Government of Tiflis) from Bulgarian roses[2]). In recent times, attempts have been made in Suchum experimental gardens to ascertain how oil supplying plants might be cultivated successfully in Batoum[3]). Already in 1899, Becklemichev had cultivated there *Eucalyptus Globulus, E. Maideni, E. amygdalina, E. viminalis, E. pulverulenta,* and other species, with a view to obtain oil from them. These experiments have not been continued, however, after his death. It resulted that the leaves picked in spring afforded the best yield in oil, which was 0.7 per cent., instead of 0.6 per cent. for *Eucalyptus Globulus,* and 2.2 per cent., instead of 1.8 per cent. for *Eucalyptus pulverulenta.* The dried leaves gave still better yields and permitted of making considerable economies with regard to fuel, the quality of the oils remaining the same. The eucalyptol contents exceeded 40 per cent. As to costs per hectare, the installation is estimated at 1200 francs, the annual expenses at 500 francs, and the yield at from 1200 to 1400 francs, so that a factory with 65 000 to 80 000 francs of capital would pay 13 to 30 per cent. of interests. Experiments to cultivate *Mentha Pulegium* (pennyroyal) failed. The yield in oil was 0.4 per cent. from fresh and 1.09 per cent. from dried herb.

Camphor leaves (from *Cinnamomum Camphora*) afforded 0.9 per cent. of crude camphor, containing 22.6 per cent. of camphor oil and 76.4 per cent. of camphor.

Bibliographical Notes.

"*Heil- und Gewürzpflanzen*" (medicinal and spice plants) is the title of a monthly paper published since July last by the Hortus society, in connection with the Munich publishing firm of J. F. Lehmann. It is intended to foster the cultivation and the collection of herbs, so as to amplify our native flora and to enable us, so to speak, to produce in our own country substitutes for all such drugs from the import of which we are cut off at present. The editors are Drs. H. Ross and R. Escales, whereas a number of other known experts are mentioned as collaborators.

Henning's book "*Der Geruch*" has been discussed on page 123 of this *Report.*

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[1]) *Pharm. Post* **1916.** As per a special reprint kindly sent us. — [2]) Comp. *Report* April **1900,** 39. — [3]) *Bullet. mens. d. rens. agric. et des maladies d. plantes* **1916,** November; *Olien en vetten* (Amsterdam) **1** (1917), 276.

Some little known essential oils. — In an article, published under this heading in the *Perfumery and Essential Oil Record* (7 [1916], 339), there is a description of about 28 little known essential oils, an in itself praiseworthy undertaking, as such products get known in this way to a large number of readers. The whole matter changes its aspect, however, when it results that the description of all the oils in question is nothing but a translation of the corresponding parts of the second edition of Gildemeister's book *Die ätherischen Öle* (The Volatile Oils).

A. Binz[1]), who critically discussed the article, expresses his opinion as follows: "Luck and ability are required for literary theft, as for all similar transgressions of the rights of other people. Otherwise one runs the risk of being found out and accused. The judgment may be left to the reader".

He reproduces the English text and the description from Gildemeister's book relating to some of the oils, and it results from the comparison that the former has been taken from the latter word for word, only various passages having been shortened, without mentioning the sources.

As Binz further mentions, all other publications are quoted, except our *Reports*. In one isolated case only (quipita wood oil), where the name of Schimmel & Co. occurred in the text, it has not been eliminated, apparently by oversight!

Reports on the progress of the chemistry of essential oils and aromatics for the period from August 1915 to July 1916 and the years 1914 to 1916 have been published by A. Reclaire[2]).

Artificial aromatics from tar products. — A. Reclaire[3]) reports on coal tar and its relations to artificial aromatics, discussing in short the preparation of the most important aromatics from coal tar and the occurrence of some aromatics or of their raw materials in said product.

Analytical Notes.

Determination of volatile oils contained in liqueurs.—According to the French Bill Ribot, liqueurs are not allowed to have a greater volatile oil content than 0.5 g. per litre. In consequence, a number of French chemists have published particulars concerning the determination of volatile oils in liqueurs which we have repeatedly referred to in our *Reports*, and at the present moment several such publications have come to hand. C. F. Muttelet[4]) declares that the only method for making such determinations quite accurately is the one which he already worked out some time ago by means of isolating and weighing[5]), in the case of the volatile oils in peppermint liqueur, because the determination of the iodine value, owing to the circumstance that only peppermint oil, but not menthol, which is frequently added for flavouring purposes, possesses a iodine number. He further mentions the determination of volatile oils in anisette, curaçao, kümmel, benedictine, chartreuse and bitter liqueurs. In the case of strongly aromatized liqueurs, in which the distillate contains volatile oil which has remained undissolved, the latter cannot be used at the same time for determining the alcohol and oil contents, on the contrary both determinations must be performed with different distillates. For the purpose of determining the oil content, 100 cc. of the liqueur are diluted in a

[1]) *Deutsche Parf.-Ztg.* **3** (1917), 14. — [2]) *Zeitschr. f. angew. Chem.* **29** (1916), I. 353. — *Chem. Ztg.* **40** (1916), 977. — [3]) *Deutsche Parf.-Ztg.* **3** (1917), 77. — [4]) *Ann. des Falsifications* **9** (1916), 70, 134; *Chem. Zentralbl.* **1916**, II. 850. — [5]) *Ann. de Chim. analyt.* **21** (1916), 50; *Report* October **1916**, 88, 89.

suitable manner with water and then distilled into a separating funnel, fitted with a mark; one then adds 50 g. of salt and 100 cc. of water, agitates first with 10 and twice with 5 cc. petroleum ether, evaporates the latter at a temperature not exceeding 30°, and then weighs. The determination of the volatile oil contents of the above-mentioned liqueurs according to the iodine value and weighing method proved that the iodine value method was only applicable in the case of aniseed and kümmel liqueurs, but that it fails in the case of the other liqueurs, whereas the weighing-method gives reliable results in the case of all liqueurs.

L. Ronnet[1]) obtained fairly good results in determining the volatile oils of aniseed and chartreuse. In his experiments, he employed alcoholic solutions of volatile oils such as are used in the manufacture of aniseed and chartreuse, and prepared the corresponding liqueurs with quantities of solution varying from 0,333 to 1.0 g. added 200 g. of sugar per litre and alcohol of 36 per cent. content. For the determination of the iodine value, he added 30 to 40 cc. of water to every 110 cc. of liqueur, distilled off the volatile components, and diluted 100 cc. of the distillate with 95 per cent. alcohol and water to 150 cc. of a liquid of 50 per cent. alcohol content. This liquid was employed for determining the iodine value by a treatment lasting 3 hours. For the purpose of calculating the content of the liqueurs in volatile oils, the coefficient 1.515 was used in the case of aniseed, and 1.498 in the case of chartreuse. The results he found proved to be in the former substance 0.113 and 0.108, in the latter 0.092 and 0.098 instead of 0.1 per cent. The same author[2]) has also examined the extracts prepared with alcohol of 18 per cent. from the plants used in the manufacture of vermouth-wine, in reference to their iodine value and found that such an extract whose preparation entailed the employment of 15 to 20 times more herbs than are generally used absorbed 1.372 g. iodine per litre. This forces one to the conclusion that vermouth-wine, prepared in the ordinary manner, can only contain an infinitesimal quantity of volatile oils.

A. Bonis[3]) recommends a combination of the iodine-value and weighing-methods which, however, is only suitable for liqueurs containing more than 0.5 per cent. of volatile oils.

R. Marcille[4]) has examined the volatile oils mostly employed in the manufacture of liqueurs in reference to their attitude when determining their iodine value and refractive power. The results obtained were as follows: The iodine value of angelica oil gave regular curves and showed an increase of the iodine number in the dark room and a reduction in full day-light. The same applies to coriander, peppermint, and balm oils. On the other hand, the iodine value curves of aniseed oil showed striking irregularities. In full day-light, they are always higher than in the dark room, besides which the curves gave a maximum in their first section, before conforming to the general impression produced by the curves of the other volatile oils. Star anise oil shows an increase of the iodine value in full day-light, just like aniseed oil. Hence it is advisable to determine the iodine number of volatile oils in the dark room and to allow the reaction to continue for a space of 12 to 24 hours.

The above results prove that all mixtures of volatile oils which indicate a higher iodine value when exposed to light than in the dark room, contain aniseed or star anise oil. The iodine value also allows one to distinguish peppermint oil from menthol, as the former does not react at all with iodine, whereas the latter has a iodine value,

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[1]) *Ann. des Falsifications* **9** (1916), 14; *Chem. Zentralbl.* **1916**, II. 856. — [2]) *Ann. des Falsifications* **9** (1916), 144; *Chem. Zentralbl.* **1916**, II. 851. — [3]) *Ann. des Falsifications* **9** (1916), 12; *Chem. Zentralbl.* **1916**, II. 850. — *Ann. des Falsifications* **9** (1916), 6; *Chem. Zentralbl.* **1916**, II. 849.

amounting to 0.58. Marcille, on the whole, regards distillation as superfluous for the purpose of determining the iodine value of liqueurs containing sugar. Some few oils, especially aniseed oil, show e reduction of the iodine value in course of time.

In any case Marcille opines that the determination of volatile oils is always a very uncertain matter in case one does not possess the oil which was employed for the production of the compound, destined for investigation.

Finally, X. Rocques[1]) refers to the papers issued by the committee formed by Rocques, Coudon, Muttelet and Ronnet. Comparative experiments were made on the volumetric method for the determination of the iodine value, recommended by Ronnet, and on the above-mentioned direct weighing method investigated by Muttelet, for the purpose of examining a number of the most generally-known liqueurs. The results can be summed up on the whole as proving that the weighing method can be universally applied, but the iodine-value method only in the case of aniseed oil, whereby the iodine value 1.450 must be taken as a basis and in the case of kümmel liqueur, the iodine value 2.400 being the basis employed.

Most of the best known brands contain more than 0.5 g. of volatile oil per litre, some few, as for instance curaçao, even more than 2 g. French vermouth contains so little volatile oil that the determination is impracticable.

Determination of the water-content of drugs. — In our last October *Report*, we referred to the difficulties encountered when determining the water-content of drugs, and which are occasioned by the circumstance that in the methods generally employed for determining moisture, such as the drying closet or the desiccator, not only the water evaporates, but also the volatile oil to a great extent or even entirely which causes the results obtained to be too high. In order to obviate this disadvantage, van Itallie, Kerbosch and Olivier[2]) recommended the determination of the moisture as such which can be effected by the distillation-method, according to which the substance to be tested is distilled with liquid paraffine, toluene, xylene, or benzine and the water which is liberated then measured. A. Scholl and R. Strohecker[1]) proceed exactly in this manner, as they distil the drugs with xylene in addition to 5 per cent. toluene and also measure the moisture which is eliminated. Thereby they arrived at just the same results as the above-mentioned authors who point out that xylene and toluene give the best results; however, neither Scholl nor Strohecker seem to have come across this paper.

On the determination of the strength of spirits of peppermint, vide page 34 of this *Report*.

A process for the determination of the oil content of sandalwood oil is described on page 58 of this *Report*.

Acenaphthene-test for distinguishing aliphatic from cyclic aldehydes. — A reaction which might be of use in examining volatile oils refers to the test described by R. de Fazi[3]), with acenaphthene (peri-ethylene naphthalene), $C_{10}H_6\begin{pmatrix}(1)\ CH_2\\ (2)\ CH_2\end{pmatrix}$, which is said to allow to distinguish aliphatic from cyclic aldehydes. He adds 1 cc. of concentrated sulphuric acid to a mixture of a few drops of a solution of the test-aldehyde in chloroform and

[1]) *Annal. de Chim. anal.* **21** (1916), 175; *Ann. des Falsifications* **9** (1916), 127; *Chem. Zentralbl.* **1916**, II. 851. — [2]) *Pharm. Weekblad* **52** (1915), 205; *Report* October **1916**, 95. — [3]) *Gazz. chim. ital.* **46** (1916), I. 334; *Chem. Zentralbl.* **1916**, II. 567. — *Journ. Soc. chem. Industry* **35** (1916), 753.

2 or 3 drops of a 1 per cent. acenaphthene solution in the same solvent; the presence of a cyclic aldehyde is then indicated by a green colouring which turns reddish-violet. This colour remains permanent for a few days and disappears on pouring the solution into water. It proved impossible to isolate the coloured substance. Benzaldehyde, salicylaldehyde, *m*-hydroxybenzaldehyde, vanilline, piperonal (heliotropine), cinnamaldehyde, cuminic aldehyde, and furfurole show this reaction. Formic and acetic aldehydes form dark-coloured addition products, without, however, producing the characteristic reddish-violet colour; ordinary aliphatic aldehydes do not react at all. Aldoses and other carbohydrates, including cellulose, which combine with cold, concentrated sulphuric acid and produce furfurole or similar aldehydes indeed show this reaction, but it lasts much longer until the characteristic reddish-violet colouring appears. It is said to be possible by this means to detect such infinitesimal quantities as 0.0078 mg. of benzaldehyde, 0.019 mg. of vanilline, and 0.006 mg. of furfurole.

de Fazi has investigated the cryoscopic behaviour of binary mixtures of acenaphthene with vanilline, *p*-nitrobenzaldehyde, and piperonal. In neither of these 3 cases was it possible to detect the formation of addition products. The melting points of the eutectics were 67.5, 66.8 and 29.9° respectively.

On the determination of vanilline in vanilla extracts, vide page 102.

On the saponification of *iso*-valeric esters, vide page 89 of this *Report*.

Some colour reactions of indole and scatole have been described on page 157 of this *Report*.

Physical Notes.

Electrical (charging) phenomena produced by the atomization of solutions of perfumes. — Perfumes offer abundant scope for investigation to the most various branches of natural science. Not only chemists but also physiologists, bacteriologists, botanists, medical men, etc. find in this branch a most productive field for their activities. That physicists can also take up the subject of perfumes with advantage is apparent from the publications of H. Zwaardemaker[1]). In company with H. R. Knoops and M. N. van der Bijl, he investigated the electrical phenomena produced by atomized perfumes as well as the spec. strength of scent and the phenomena of odoroscopic charging in homologous series, besides the possibility of charging mixtures of perfumes by means of secondary electrical currents. Zwaardemaker employs a larger atomizer for the purpose of producing a mixture of atomized perfume and air, such as was built some years ago by G. Gradenigo[2]). If we atomize a watery solution of non-volatile substances with this apparatus, one obtains a fairly stable kind of mist, watery solutions of perfumes, however, form a mist which disappears already in a few minutes. By means of an ultramicroscope one can observe small drops of water in the mist and active Brownian motion. The scent is even observable after the mist has disappeared which proves that perfumes act on the nasal organs not only in the shape of mists but also in a gaseous state[3]).

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[1]) *Koninkl. Akademie van Wetenschappen te Amsterdam* **24** (1916), 1630; **25** (1916), 3, 512. (Meetings of March 25th, May 27th and September 30th.) According to separate impressions kindly forwarded to us. Other publications by the same author have formerly been discussed by us. Vide *Report* October **1904**, 104; October **1908**, 160. — [2]) A description of this apparatus is to be found in the *Arch. int. de laryngologie, d'otologie et de rhinologie* **1911**. Vide also H. Zwaardemaker, *Arch. Néerland. de Physiol. de l'homme et des animaux* **1** (1917), 347. — [3]) J. Gaule expressed, in 1900, the idea that perfumes in Nature occur generally in the form of mists or vapours and that they are only perceptible to us in that state.

If one atomizes water or a solution of salt and directs the vapour against a metal plate which has been carefully isolated by amber and is connected with an electroscope, the latter remains at rest. On the contrary, if one atomizes a solution of a perfume, the metal plate becomes positively charged when an atomizing pressure of 2 atmospheres is employed. Whereas the vapour vanishes, on interrupting the experiment, the electroscope, however, remains charged. Under the experimental conditions employed by Zwaardemaker even the small drops of water which run off the metal plate retain a positive charge. Zwaardemaker tested a great number of perfumes and of substances having a strong odour in reference to their capacity for becoming charged electrically when in a form of vapour; he obtained satisfactory results with acetaldehyde, acetone, ether, ethyl alcohol, ethyl bisulphide, ethyl bromide, ethyl butyrate, ethyl malonic acid, allyl sulphide, formic acid, ammonia, amyl acetate, amyl alcohol, amyl butyrate, anethole, aniline, anise aldehyde, methyl anthranilate, apiol, valeric acid, benzaldehyde, borneol, bromine, bromoform, carvone, quinoline, chloroform, citral, citronellol, cumene, decylic aldehyde, duodecylic aldehyde, acetic acid, eucalyptole, eugenol, formic aldehyde, guaiacol, heliotropine, *iso*butyl alcohol, irone, iodine, ionone, linalool, menthol, mercaptane, methyl butyrate, methyl salicylate, myrtole, naphthalene, nonyl aldehyde, paraldehyde, light petroleum, propylamine, pulegone, pyridine, safrole, scatole, styrone, thymol, trimethylamine, undecylic aldehyde, vanilline, xylene, and cinnamaldehyde. Amongst genuine perfumes no exception whatever were observed. Substances like ethyl malonic acid, benzaldehyde, camphor, citral, eugenol, geraniol, heliotropine, ionone, menthol, trinitrobutyltoluene are charged electrically even in the most diluted solutions, others only slightly, even very little indeed, which refers especially to ammonia. Chlorine water or such containing ozone, produced no perceptible charge.

If one employs a screem of metallic gauze instead of a plate, the former is charged positively, whereas the particles which are driven through the screen take on a negative charge; they can also be collected by means of a metal plate placed behind the screen. The little drops which flow off the gauze have a much stronger odour than those collected on the second screen. Strange to say, a 2 per cent. alcoholic solution also produces when atomized a perceptible charge; the latter, however, becomes weaker and weaker if the concentration is increased, and when a concentration of 50 per cent. is used, their is no charge whatever. The same applies to acetone, pyridine, and other substances.

Surprisingly small quantities of perfumes are sufficient to produce electrical charges. $25 \cdot 10^{-6}$ g. of geraniol, dissolved in 25 cc. of a 2 per cent. sodium salicylate solution, suffices to plainly produce an electrical charge. The same applies to the same quantity of trinitro butyl toluene. Our nasal organs are, however, much more susceptible and can perceive even much smaller quantities; yet it is by no means impossible that if a very sensitive electroscope is employed much smaller quantities of perfumes might be detected.

Perhaps the vapour-electricity-method, might be employed for practical purposes, when the question arises of proving if a perfume is at all soluble in water or not.

Probably the molecular weight, volatility, and the reduction of the surface tension are the factors which determine the quantity of a substance which is required to produce the electrical charging phenomena. These are the same requirements to which a compound has to conform in order to act as a perfume from a biological point of view: Hence a connection between the intensity of the odour and that of the charge phenomena is to be expected, a manifestation which will be most apparent in homo-

logous series. J. Passy[1]) has referred to this fact in his notes on the intensity of the odour of aliphatic alcohols. He applies the term *minimum perceptibile* to the smallest quantity contained in one litre of air which can still be smelt. If one divides the value of this quantity expressed in grams by the molecular weight of the substance and the result again into 1, one obtains the spec. intensity of smell. For the above-mentioned alcohols and acids one finds under these conditions the following values: —

Table I. *Intensity of odour of aliphatic alcohols.*

	Minimum perceptibile expr. in grs. per litre.	Molecular weight.	Specif. odour intensity.	Log. mol. intensity of odour.
Methylalkohol	$1000 \cdot 10^{-6}$	32	$0.032 \cdot 10^{6}$	0.51
Ethylalkohol	$250 \cdot 10^{-6}$	46	$0.18 \cdot 10^{6}$	1.26
Propylalkohol	10 to $5 \cdot 10^{-6}$	60	$6 \cdot 10^{6}$	2.78
Butylalkohol	$1 \cdot 10^{-6}$	74	$74 \cdot 10^{6}$	3.87
*Iso*amylalkohol	$0{,}1 \cdot 10^{-6}$	88	$880 \cdot 10^{6}$	4.94

Table II. *Intensity of odour of fatty acids.*

	Minimum perceptibile expr. in grs. per litre.	Molecular weight.	Spec. odour intensity.	Log. mol. intensity of odour.
Formic acid	$25 \cdot 10^{-6}$	46	$18 \cdot 10^{6}$	0.26
Acetic acid	$5 \cdot 10^{-6}$	60	$120 \cdot 10^{6}$	1.08
Propionic acid	$0.05 \cdot 10^{-6}$	74	$1480 \cdot 10^{6}$	3.17
Butyric acid	$0.001 \cdot 10^{-6}$	88	$88000 \cdot 10^{6}$	4.94
Valeric acid	$0.01 \cdot 10^{-6}$	102	$10200 \cdot 10^{6}$	4.01
Caproic acid	$0.04 \cdot 10^{-6}$	116	$2900 \cdot 10^{6}$	3.46
Œnanthylic acid	$0.3 \cdot 10^{-6}$	130	$430 \cdot 10^{6}$	2.63
Caprylic acid	$0.05 \cdot 10^{-6}$	144	$2880 \cdot 10^{6}$	3.46

If one plots out the mol. weights on to the abscissa and the mol. odour intensities on to the ordinata of a coordinate system, one obtains exceedingly regular lines.

Even the charge capacity of the vapours can be demonstrated. If one places a tin disc at a distance of 1 to 2 mm. in front of the atomizer, the former remains uncharged. If one removes it, however, from the atomizer, its charge grows in a direct ratio to the distance, up to a certain maximum; when once this has been surpassed, the charge diminishes again and finally disappears altogether at a certain distance which is characteristic for every substance. Zwaardemaker calls this distance the critical one.

Table III. *Critical distance for aliphatic alcohols expressed in centimeters.*

Substance.	0.3 normal.	0.2 normal.	0.1 normal.
Methylalkohol	46	26	3
Ethylalkohol	103	70	35
Propylalkohol	145	80	72
Butylalkohol	170	100	117
*Iso*amylalkohol	187	135	130

One obtains a graphical representation of the tables I and III if one plots out the values of the molecular weights on to the axis of the abscissa and the logarithm of the values of the spec. odour intensities and the critical distances on to the ordinates.

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[1]) *Compt. rend.* **114** (1892), 306, 1140; **116** (1893), 1007.

In this manner one obtains lines running almost parallel to each other. The same applies to the table II and IV; in this case the lines run in a different manner, but in the same sense.

Table IV. *Critical distances for fatty acids expressed in centimeters.*

Substance.	1/10 n.	1/20 n.	1/40 n.	1/80 n.	1/160 n.	1/320 n.	1/640 n.
Formic acid	1.2	0	—	—	—	—	—
Acetic acid	20	0.5	—	—	—	—	2
Propionic acid	81	50	—	—	—	—	20
Butyric acid	125	85	—	—	—	—	74
*(Iso)*Valeric acid	170	173	—	—	—	—	78
Caproic acid	—	190	180	177	160	132	106
Caprylic acid	—	—	—	—	—	—	90

Experiments with mixtures have proved that the charging phenomena in this case are additive; accordingly mixtures which have an exceedingly faint or scarcely perceptible odour in which, therefore, a considerable scent-compensation has taken place, can — owing to this additive quality — show an increased charging capacity.

The results are quite different in cases in which the components of a mixture react chemically on each other, for instance with compounds such as aniline and acetic acid which produce acetanilide. If under this circumstances a compound is to produce an electrical charging phenomenon, it must conform to the following conditions:—

1. It must be soluble in water (compounds which are totally insoluble in water do not show this phenomenon at all);
2. It must reduce the surface tension of water.
3. It must, when finely divided over a great surface, become volatile from the solution.

Acetanilide conforms to the conditions 1 and 3 and really belongs to the strange group of organic compounds which produce the charging phenomena, just like perfumes themselves.

If the compounds which conform to the above-mentioned 3 conditions are also perfumes by nature, they must be able to adapt themselves to two further ones, namely:—

4. of being soluble in lipoids (all perfumes are soluble in fatty oils)
5. of containing an odorophorous group in their composition.

Acetanilide hardly conforms to the condition N° 4, but the aniline and acid radicles can be regarded as forming an odorophorous group. Rupe and von Majewski[1]) suspect that, in case a molecule contains 2 odorophorous groups possibly they tend to neutralize each other in their effects. This fact, besides its insufficient solubility in oil, might explain the odourlessness which is a characteristic of acetanilide.

Besides the above-mentioned publications, papers by E. L. Backman[2]) on the olfactologie of the methylbenzene series and by C. Huijer[3]) on the olfactologie of aniline and its homologues. Backman has found *inter alia* that the *minimum perceptibile* of the perfumes in the methylbenzene series, in so far as it applies to the first 4 links, (benzene, toluene, xylene and pseudocumene), regularly diminishes with the increase in the number of the methyl-groups. However, the intensity of scent increases under these circumstances first of all. The odour turns from an empyreumatic one (benzene — toluene) to an aromatic one (xylene — pseudocumene) and then into a phenolic one (durene).

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[1]) K. von Majewski, *Beiträge zur Kenntnis der Diazoimidobenzolderivate.* Inaug.-Dissert., Basel 1898. — [2]) *Koninkl. Akad. van Wetensch., Amsterdam* **25** (1917), 971; meeting of Jan. 27th. According to a kindly forwarded referate impression. — [3]) *De olfactologie van aniline en homologen.* Inaug.-Dissert., Utrecht 1917. According to a copy kindly forwarded.

At the same time its intensity diminishes, so that possibly odourless compounds might be formed if the methylation were pushed far enough. Huijer has ascertained concerning the aniline series that the *minimum perceptibile* diminishes for the higher links and that the intensity of odour increases. The smell of the 3 isomeric toluidines varies; *o*-toluidine has the least, *p*-toluidine the strongest smell. When equimolecular solutions of these are atomized the electrical charge shows a great increase in the case of the higher links of the series, and of these 3 *o*-toluidine produces the smallest and *p*-toluidine the greatest charge.

The maximum tension of the vapour of pure camphor.—Two methods are in use for the determination of the maximum tension of vapours, *viz.*, the barometric method and the evaporation method. P. Datin[1]) has compared both in relation to their reliability by determining the maximum tension of the vapour of pure camphor. The barometrical method gave the following results (in mm.): 0.46 (18.9°), 0.55 (23.4°), 0.94 (28.4°), 1.04 (30.8°), 1.32 (36°), 1.70 (40.2°), 2.22 (47°), 2.72 (52.5°), 3.12 (55°), 4.22 (62°), 5.61 (66.6°). The evaporative method (passing a gas current slowly over a weighed quantity of substance and determination of the loss in weight) gave the following results: 0.39 (18.2°), 0.53 (23.4°), 1.15 (34.2°), 1.71 (40.3°), 2.24 (48.6°), 3.25 (57.8°), 8.66 (77.5°). Accordingly, both methods lead to results which agree satisfactorily. The evaporative method is to be preferred when it is a question of determining *low* pressures and in point of fact is more suitable for maximum pressures which are lower than 2 mm.; however, it is only really reliable when the air current is passed over for a considerable time.

Botanical Notes.

Sweet-smelling plants from German South-west Africa.—Some years ago we discussed a paper by G. Volkens[2]) on the valuable plants growing in Togo, in which the author has also referred to a number of sweet-smelling ones. Those who care for further information on the aromatic plants of our colonies will find the some particulars in a publication by K. Dinter[3]) on sweet-smelling plants from German South-west Africa. Of course, the author only makes suggestions, and concerning many plants it will be a doubtful matter if the aroma can be easily obtained and if it would be worth while. Dinter believes that most of the plants he discusses could be easily cultivated.

He enumerates first of all the blossoms of some species of acacia, especially because they might most easily be collected in quantities. In point of fact, all of the 28 species which have hitherto been known to originate from this country have an odour, and none of them an unpleasant one, but in no case do any of them even approach the violet-like sweet odour of the camel-tree *Acacia-Giraffæ*, whose delicious scent quite pervades the air of the valleys which bear groundwater at the beginning of the dry and hot early summer, *i. e.* in September. *Acacia horrida* has a similar, but not so purely violet-like smell. The *Ana*-tree, *Acacia albida*, blossoms towards the end of May, already in the cold season, and its odour reminds most of limeblossom. The same applies to *Acacia hebeclada* which blossoms in September.

The blossoms of the Thymelacea, *Gnidia polycephala* which flower in midwinter in Okahandja possess a wonderfully sweet smell like orange flowers.

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[1]) *Annal. de Phys.* IX. 5 (1917), 218; *Chem. Zentralbl.* **1916**, II. 1915. — [2]) *Notizblatt des Kgl. Bot. Gartens und Museums zu Berlin-Dahlem, Appendix* XXII. 3, (Nov. 30th 1910), 70; *Report* April **1911**, 144. — [3]) *Deutsche Parf.-Ztg.* **3** (1917), 53.

A wine-like odour is characteristic for the little clusters of blossoms borne by the little Capparidacea-tree, *Boscia Pechuelii* which flower in September and are quite common all over the Herero- and Nama-lands; on the contrary, the blossoms of *Boscia fœtida* are distinguished especially at a certain distance by an awful smell of human excrements. On the other hand, the beautiful flowers of the 13 species belonging to the genus *Cleome*, N. O. *Capparidaceæ*, are quite odourless, whereas the leaves of some of these species smell exceedingly strongly aromatically.

The white spiraea-like clusters of blossoms of the Aizoacea-bush *Anisostigma Schenckii* which occurs in the brackish sand and clay formations of Nama-land smell of mignonette.

A strawberry-like odour is characteristic for the flowers of the Capparidacea *Maerua Gürichii*.

Cephalocroton Püschelii, an Euphorbiacea growing up to the height of 1 m. has blossoms which remind exactly of the smell of violets.

The transparent white bell-like buds of the Amaryllidacea *Pancratium Chapmani* have the property of bursting after the first heavy rains in December and January between 5 and 7 in the evening and then develop a very strong smell of hyacinths, which, however, only lasts until about 7 o'clock the next morning. The buds then remain completely closed. It is a very striking fact that the numerous South-west African Liliaceæ have no odour at all, whereas the equally common Amaryllidaceæ nearly all are distinguished by a weak or sometimes very strong lily-like smell which depends on the species they belong to. The genus *Crinum* is especially worth mentioning in this respect.

The odour of the Rubicacea *Gardenia Thunbergia* can hardly be considered inferior to that of *Gardenia floribunda*.

The Sterculiacea *Hermannia atrosanguinea* and similar species smell of good hock.

Jasminum mauritianum has the same odour as the other white-flowering jasmine species.

The odour of the Iridacea *Lapeyrousia stenoloba* is exactly like that of the well-known *Freesia*.

The Polygalacea *Securidaca longepedunculata*, a tree which grows to the height of 8 m., bears clusters of blossoms, smelling of violets and has hence been named "violet-tree".

The scent of the greenish-white blossoms of the Liliacea *Sansevira thyrsiflora* is just like that of hyacinths.

The blossoms of the Aizoacea *Mesembryanthemum inachebense* have a pleasant odour; all the other 57 *Mesembryanthemum* species in South-west Africa are odourless.

Numerous plants in South-west Africa are distinguished by an exceedingly unpleasant smell, for instance *Boscia fœtida*, which has already been referred to; the same applies to the *Hydnora* species (N. O. *Hydnoraceæ*), root parasites whose blossoms smell of carrion and whose long and wide flower-tubes are hence much frequented by numerous small dung-beetles and fly-larvæ. Some species of the genus *Stapelia (Stapeliaceæ)* have an awful odour of carrion, others, as for instance *S. Fleckii*, smell strongly of honey.

The Compositæ *Peschuel-Löschea Leubnitziæ* and *Blumea gariepina*, the Geraniaceæ *Monsonia umbellata* and *senegalensis*, as well as the Euphorbiacea *Croton gratissimus* contain volatile oils[1]). *Blumea gariepina* smells strongly of dill, *Pechuel-Löschea* "just like niggers". *Helichrysum cerastoides* has a strong odour of licorice. The following are also distinguished by strong scents: —

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[1]) *Report* October **1913**, 111.

Compositæ Helichrysum benguellense, Matricaria albida and *globifera, Osteospermum muricatum* (of apples), *Homochæte forulosa.*

Scrophulariaceæ: Chænostoma, Sutera, Lyperia, Manulea, Selago Nachtigalii and *S. alopecuroides* (both strongly of garlic).

Labiatæ: Ocimum canum[1]), *Plectranthus, Moschosmia.*

Leguminosæ: Psoralea obtusifolia (used as a substitute for tea by the Hereros), *Hoffmanseggia rubra* (unpleasant odour of tar), *Copaifera Mopane.*

Rutaceæ: Thamnosma africanum (has a most disgusting odour).

Moringaceæ: Moringa ovalifoliata.

Umbelliferæ: Peucedanum fraxinifolium (the bark is much appreciated by cliff-badgers owing to its aromatic taste).

Myrothamnaceæ: Myrothamnus flabellifolius. The leaves are much in favour amongst the Hereros for making tea.

Euphorbiceæ: Excœcaria africana (the wood is much appreciated by the Hereros for the purpose of making beads for necklaces).

Liliaceæ: Tulbaghia calcarea and *T. tenuior* (both smell of garlic).

Cyperaceæ: Kyllingia alba, whose root fronts are much in favour among the Hereros owing to their slight smell.

Cucurbitaceæ: Kedrostis fœtidissima, when burnt, produces a most pestilential, undefinable odour.

The fragrant plants of Asia Minor. — K. Krause[2]) has discussed the fragrant plants of this country as a continuation of his paper on the prospects of the cultivation of aromatic plants in Asia Minor[3]). Climatic conditions quite exclude the existence and cultivation of aromatic plants in the North and in the interior of Asia Minor and both are only possible in the West and South. The original flora is found here in the shape of those characteristic evergreen bushes which in Italian are termed "macchia". Those of this country hardly differ at all from the ones occurring in the other country Mediterranean regions. Just like these, they consist mainly of dense, evergreen, hard-leaved bushes which often start growing quite close by the sea from which they are frequently only seperated by a narrow strip of sand, hardly wider than a few yards. In the interior, they are gradually transformed at a height of 300 and 400 m. into the deciduous leafy woods which cover the upper precipices. In some valleys, they grow to an altitude of 600 m. or higher, but in such cases they are mostly intermixed with other plants. Besides *Quercus coccifera* and *Q. ilex* (stone oak) the following bushes abound in the "macchia" of Asia Minor: juniper *(Juniperus oxycedrus),* jasmine *(Jasminum fructicans),* pistachio nuts *(Pistacia terebinthus* and *P. lentiscus),* laurel *(Laurus nobilis),* myrtle *(Myrtus communis),* various species of the genus Cistus (especially *Cistus villosus, C. laurifolius, C. salviifolius, C. creticus),* and many others. The characteristic odour of the "macchia" is even observable at a distance when there is no wind. Some species, amongst others juniper *(Juniperus oxycedrus),* as well as several citrosæ are so abundant that they quite predominate and one accordingly terms the "macchia" the juniper or "citrosæ-macchia". In the lowlands of Western Asia minor, another plant formation is superabundant which is called "Phrygana" and which mainly consists of low-growing bushes, scrub and of lingnified bushes amongst which, however, aromatic plants are comparatively rare.

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[1]) *Report* April **1914**, 72. — [2]) *Deutsche Parf.-Ztg.* **2** (1916), 273, 299. — [3]) *Deutsche Parf.-Ztg.* **2** (1916), 241; *Report* October **1916**, 81.

Frequently the macchia and phrygana are pierced by glades in which numerous bushes and herbs have taken root and which are particularly rich in the sweet-smelling varieties of the species *Rubaceæ* and *Labiatæ*. In the same manner as on the Riviera one observes extensive surfaces in Western and Southern Asia Minor covered by lavender-bushes and species of sage and rue. *Lavandula Stœchas* is especially frequent. In the dry beds of rivers and streams, oleander *(Vitex agnus castus)*, tamarisks, etc. are to be found.

Krause mentions myrtles as the most important aromatic bushes of Asia Minor. In the interior and in the North, it is quite absent.

In spite of its being so common it is hardly utilized at all in most parts of Asia Minor. The Turkish term for it is "mersin" or "mirsin" and the name of the town of Mersina gives proof of its occurrence there. The laurel bush (Turkish *"dafnê"* or *"defnê"*) has about the same range of dissemination as myrtles have.

Roses which the Turks, being great lovers of nature and horticulturalists, cultivate everywhere [1]), are not used for the preparation of rose oil or rose water, but rather for the manufacture of a kind of sweet-meat.

Jasmine *(Jasminum officinale)* is much less frequently cultivated as a garden plant than roses.

Lemons, oranges and bitter oranges are regarded as much more important objects for cultivation.

Lavender has been collected in Asia Minor from time immemorial and in the height of summer in the streets and squares one often sees the lavender sellers laden with big baskets in which they offer their produce for sale, tied up in bunshes or bundles.

On the *Phoma*-disease of lavender, *vide* page 27.

On a new *Styrax*-species, *S. Siamensis*, Rordorf, *vide* page 89 of this *Report*.

On the botanical investigations of the vanilla-species found on Tahiti, *vide* page 101 of this *Report*.

Phyto-physiological Notes.

Experimental investigations on the physiological and biological importance of volatile oils and flower scents. — Although we do not yet know what physiological and biological importance must be ascribed to volatile oils and flower scents, some ideas have indeed been ventilated on this subject; however, they have been found in the main to be erroneous. Such suppositions are that volatile oils protect plants from animal attacks, or from too great loss of temperature at night, or from excessive evaporation during the day, or that the strong odour attracts insects, &c. K. von Frisch [2]) has now arrived at the conclusion, based on his most recent investigations, that neither the scent nor the coulour of the plants can be regarded as enticement, but rather as guidance. It goes without saying that the scent of a bush in full bloom or of a whole field of sweet-smelling flowers may attract bees from a distance, especially when the wind is favourable, but the determing factor is not the *odour* of the plants, but their *colour*. In one respect, however, the former is superior to the latter, namely in its greater variety.

Von Frisch employed for his experiments little cardboard boxes (measuring 10×10×10 cm.) which at the front just above the bottom were fitted up with a hole

[1]) *Vide* p. 47 of this *Report*. — [2]) *Verhandlungen der k. k. zoologisch-botanischen Gesellschaft in Wien* **65** (1915), *Heft* 1 *bis* 2; *Naturwissenschaftliche Wochenschrift* **31** (1916), 250.

of 1½ cm. diameter. The covers of the boxes could be opened. Two of the 4 boxes which were placed in readiness always remained empty. The rest were filled with any odour, mostly a sweet-smelling one which most resembled the scent of natural flowers and was always available in large quantities. The boxes besides contained a little receptacle filled with sugar-water which served as nourishment for the bees after being previously attracted by honey. In order to avoid training them to visit the same spot always the relative positon of the boxes was frequently altered. The question to be decided was now if bees are capable of perceiving the scent and secondly if they realize the correlation between scent and food. For this purpose after the training process just described, all the boxes were removed and replaced by four new ones which had not been used. One of these contained the scent. Hereupon the bees were counted which entered each box.

The experiment proved that the bees sought for the accustomed food in the *scentet* boxes even when it was absent. In order to determine if bees are capable of finding out a certain scent amongst various other ones, they were provided with food for some time in boxes scented with the odour of acacias. Hereupon the training boxes were substituted by others which had hitherto not been used and of which one was filled with the aroma of acacia, one with that of roses, and one with lavender scent, whereas the fourth box remained umscented. The result was that the bees all congregated in the box scented with acacia-aroma.

These and similar experiments proved that bees are capable of making a fine distinction between various odours.

The author further tried to determine if colours or scents were more observed by bees when both factors competed with each other. For this purpose, he employed a little box painted blue in front and filled with scent and sugar water, and another one painted yellow which, however, remained empty. He then trained the bees to visit the blue, scented box. After this, both boxes, were removed and replaced by an unscented blue one and a yellow one filled with scent. The bees showed no marked preference for either box, but visited both after some hesitation. Only quite at the beginning of the experiment was a marked preference observable, as apparently the bees were able to distinguish colours at a much greater distance than scents, as they flew straight to the blue box from a distance of several meters and only became perplexed, when quite near to it, because they then noticed the absence of the accustomed odour. In spite of the fact, therefore, that the behaviour of the bees varies with different scents and that the direction of the wind is not without effect in this matter, evidently both scent and colour have a combined effect.

It is especially remarkable that bees can also be trained to frequent disagreeable odours, as for instauce that of lysol, as soon as they perceive that the latter is identical with a source of nourishment.

In a paper, entitled "Volatile oils from lichens" Schelenz[1]) refers to the already known distillates of *Cladonia rangiferina* and of other cryptogamous plants, without, however, adducing any new facts, so that it is sufficient just to point out this publication.

Occurrence of anemonine in Caltha palustris. — Anemonine which is found in some species of *Ranunculaceæ*, also occurs, according to E. Poulsson[2]), in the marsh-marigold *(Caltha palustris,* L., N. O. *Ranunculaceæ).* He worked up considerable quantities of

[1]) *Pharm. Zentralbl.* 58 (1917), 59. — [2]) Comp. *Report* April 1915, 79.

this plant and proved that cases of poisoning by *Caltha palustris* must be ascribed to its anemonine content. Alcaloids were totally absent, he found, however, considerable quantities of choline. Former statements concerning the presence of alcaloids in *Caltha* must be explained by the respective authors having confounded them with anemonine.

The smell of oil of bitter almonds, emanated by Psalliota campestris.—On page 100 of our October *Report* **1916**, we discussed a paper by E. Herrmann[1]) on the odour of fungi. The development of the sense of smell is a very individual matter and some people, especially those who have not much experience therein, have great difficulty in detecting any other odour connected with fungi than the ordinary "mushroom-smell". One must add to this the fact that a characteristic odour of any kind is very hard to describe if it is possible at all. In spite of this it is a very striking circumstance that most, even experienced fungi-experts are not aware of the fact that the mushroom *par excellence* (French champignon, *Psalliota campestris* and *arvensis*), often has a characteristic odour of oil of bitter almonds, as R. Kobert[2]) mentions. For many years past he has been in the habit of carefully smelling all edible mushrooms he or his wife collected or bought, when they cut them up, and he has found thereby that the *Psalliota* species, independantly of the special variety they belong to, can be divided into two groups, namely those smelling of aniseed and those with the odour of oil of bitter almonds. Whereas most books on fungi mention the smell of aniseed, the odour of bitter almonds was totally unknown to all experienced fungiologists, and the only book which refers to this fact is the one by E. Gramberg[3]) which indeed mentions it in reference to *Psalliota campestris* and *arvensis*. Kobert has even been able to detect the scent of bitter almonds when he distilled specially assorted mushrooms which he used for purposes of cultivation, and refers to this matter in his books. A dish of fungi in which even only *one* such special mushroom is present, generally has a taste of bitter almonds. According to Kobert the smell of bitter almonds is the most essential characteristic for distinguishing a genuine mushroom from a *Knollenblätterschwamm*. In a well-known book on fungi[4]) the odour of *Psalliota campestris*, that of *P. arvensis* and that of a cultivated variety is only referred to as "pleasant". Other books on fungi use the same term and special mention is made of the aniseed odour, or no reference at all occurs concerning any special smell. In any case the data concerning the odours of fungi in most books on fungi, in botanical and other handbooks are strangely incomplete.

Physico-pharmacological Notes.

Psychological studies on odours.—In our last *Report*[5]) we discussed H. Henning's studies on odours. These papers which were formerly issued in the *Zeitschrift für Psychologie* as separate essays have now been collected as well as their continuations and published in book form[6]).

In connection with our last year's abstract, we now refer to the further results Henning obtained. His investigations place it beyond any doubt that the smell of any chemical compound is a constitutional characteristic of matter in general.

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[1]) *Tidsskrift for Kemi, Farmaci og Terapi* **1916**, 237, 262; *Apotheker Ztg.* **31** (1916), 472. — [2]) *Pharm. Zentralh.* **56** (1916), 555. — [3]) *Schmeils naturwissenschaftliche Atlanten. Pilze der Heimat. Eine Auswahl der verbreitetsten Pilze in Wort und Bild von Gramberg.* 2 vols. Leipzig 1913. — [4]) E. Michael, *"Führer für Pilzfreunde"*, vol. I. Zwickau 1903. — [5]) *Report* October **1916**, 103. — [6]) H. Henning, *Der Geruch.* Leipzig 1916.

Henning refers to the fact that scent-producing groups, ("osmophores"), owing to the possibility of their being mutually exchanged without a fundamental alteration of their odour, (for instance nitrobenzene and benzaldehyde), cannot be the *sole* determinants in producing the smell of any compound, but rather that the whole manner in which these groups are combined in the molecule is the deciding factor in producing odours. There is no doubt whatever that the osmophoric group must combine with some „osmogene" radicle or other in order to produce a smell of any kind. In this case, the smell depends on the manner the osmophores are combined with the osmogene nucleus.

Henning draws the conclusion from this consideration that the formulæ of the chemical representatives of his 6 odour-classes (*vide* his odour-prism)[1]) must—within each psychological division—have something in common as to the manner they are linked up to each other, and in point of fact without depending upon the circumstance to which chemical family these perfumes may belong to.

This hypothesis was confirmed on investigating all aromatic compounds whose constitutional formula was known. The hexagon benzene nucleus is employed as a fundamental model of the various odours. Under these circumstances, the formulæ of the perfumes belonging to one and the same psychological odour-class, for instance to the aromatic class, are linked up in the same manner. The characteristic of the aromatic scents would then be that there osmophoric groups are in a para position to each other.

The odour-prism proves that, for instance, anise aldehyde, anethole, chavicole, methyl chavicole, *p*-cresol methylether which all are para derivatives belong to the class of the spicy scents. The model is, however, also applicable to bodies of the aliphatic series if one regards them as an open hexagon with lateral chains. This structure applies to many fruit-scents, for instance, to linalool, citral, and to methylheptenone. We must restrict ourselves here only to giving some indications of the scope of this theory, as the author has reserved the right to expound it more fully in a chemical review; however, it is doubtful if it can lay a claim to general application in its present form, because—in order to give only *one* instance—cinnamaldehyde does not show the para position, demanded by Henning, although odoro-psychological test places it beyond doubt in the class of the spicy scents.

In reference to the physical nature of odours, the conception was current already in ancient times that perfumes emanate material particles which are perceived by the nasal organs, and this is the so-called "corpuscular theory". The "wave theory" of odours which was in vogue at one time can be regarded as decidedly antiquated. Our present knowledge of the physical and chemical qualities of smelling compounds gives the palm to the former theory. This supposition is also supported by physiological and psychological facts.

Another question which has been much discussed is the problem in what manner one can most accurately measure any odour. In spite of many experiments, no method has hitherto been evolved for doing this in a faultless manner. After discussing the various methods in use hitherto for this purpose, Henning describes an apparatus, consisting of a modification of the oldest method conceived by Valentin, and which is based on the determination of weight.

However, the measurement according to the volumetric method has proved to be more convenient, and for this object he describes an apparatus which has been adapted from vessels employed for gas-measuring purposes.

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[1]) *Bericht* **1916**, 114.

The measurements performed with this apparatus gave the following results:—

1. The *minimum perceptibile* is generally perceived quicker and with less smelling material in cases in which the test-person is conscious as to what is going on than *vice versâ*.
2. Test-persons observe scents which are known to them more readily and with less waste of perfume than otherwise.
3. Practice has great influence on the results.
4. Central factors and experience have also a great influence on the results.
5. During the first moments in which one begins to smell anything (the borderland of sensation), the perception is still vague, *i. e.* it is undeterminable if the scent in question appeals to the nasal organs or to those of taste or touch.
6. In order to entirely perceive any odour and to be able to characterize it (borderland of realization), both time and a further amount of perfume are required beyond that necessary for mere perception. Well-known odours are more readily perceived and also in lesser quantities than unknown ones.
7. A state of excitement or a too vivid play of the imagination tend to prolong the time required considerably.
8. The excellence and keenness of the sense of smell is not influenced at all by sexual differences. In comparing the two it was proved that *experience* was the deciding factor.
9. Children showed no difference in their detection of odours which they knew in comparison to adults. However, they are more easy to lead astray by suggestion.
10. The volume of the nasal space has no influence on the results.

A table is then adduced in which it is shown by a great number of scents how many millionths of a gram of scent per litre of air suffice in order to produce a perceptible effect, which allows one to conclude how great the absolute quantity of scent is contained in one respiration. As, however, only a fraction of the scented particles reach the nasal mucous membrane, one can estimate that although barely a single molecule of a strong perfume, for instance of musk, does not suffice to produce a stimulus, a sharp nose, however, can already detect the trillionth part of a gram. Some perfumes are so strong that a single gram would be sufficient in order to produce a perceptible sensation to all the inhabitants of the earth (1600 millions). Hence the nasal organs are a hundred-thousand times more sensitive than the methods of spectral analysis. There is, therefore, no doubt that of all our senses that of smell is the most sensitive.

According to Henning's observations, the much discussed question if human beings are capable of smelling liquid solutions of scents must be answered in a negative sense. The sensations produced in such cases are not really those of smell, but rather tactile ones, combined with those of taste and temperature.

Experiments with aquatic animals prove that they are indeed capable of smelling aromatic substances dissolved in water, but that their smelling sensations are mainly affected by the non ionized scent particles which are diffused in water without being dissolved. The physiological process of smelling in human beings can be thus explained that the molecules of perfume diffused in the air penetrate the mucous membrane and are split up on the surface of the individual smelling cells. The intensity of the sensation seems to depend on the greater or lesser number of scented molecules which act together.

Opinions differ still widely as to the efficiency of the human sense of smell as well as its meaning in biological, phylogenetical, physiological, psychological, and æsthetic respect.

Doubtless many animals have a keener sense of smell than human beings; however, this does not justify the saying that the human smelling organs are by any means degenerated. No substance exists which human beings are incapable of smelling — provided it is sufficiently concentrated — but most people are simply inexperienced in this matter. Extraordinary results can be attained by practice. But no doubt many human beings suffer from diseases of the nasal organs, probably in consequence of the habit of eating food too rich in albuminous compounds.

Formerly, a perfectly fabulous sense of smell was ascribed to some primitive nations, and people imagined then capable of detecting and pursuing human tracks by its aid. However, nowadays one has come to the conclusion that in such cases the sense of sight is the predomimant factor.

That each race has a peculiar odour is generally acknowledged at the present day, and there is no doubt of the fact that the various races object to each other's odour.

The customary nose-greeting found amongst some primitive nations must also be ascribed to the race-odour; such peculiarities can find expression in their languages, as for instance some of such nations salute each other with the words: "smell me" or "let's rub noses."

It is worth remarking concerning the development of the sense of smell in children that such sensations are already marked even within the first days after birth.

The æsthetical importance of perfumes is generally known; the oldest records of the history of civilization already give testimony to the value placed on them.

As the capacity of the normal sense of smell can be vastly increased by practice, one ought to pay more attention to its development during education.

The effect of scents on animals has frequently been studied. There is no proof whatever in this case of a specifically chemical sense, although some authors claim to have observed it. In the lowest organisms, a certain general irritability can be taken for granted as the first form of the sense of smell and shows itself equally in the attitude of the plasm of bacteria and of unicellular beings towards osmosis, electricity, temperature, light, pressure, gravity, and chemicel influences. This vague primitive irritability was certainly the foundation from which the various senses gradually were developed, but there is no advantage whatever in regarding this primitive irritability as a special sense.

On the whole, we do not find that the lowest organisms, like bacteria for instance, plainly react in a positive sense towards useful and nutritious substances and in a negative sense towards noxious and deadly ones; on the contrary, strange and hitherto unexplained differences in their attitude are apparent.

A specific sense of taste or smell does not exist amongst them.

That the coelenterates have a sense of smell can be demonstrated. Worms also show well-defined smelling organs which may be detected in the parts about the head and already surpass the rudimentary forms required for distinguishing food. The echinoderms, mollusks and crustaceæ also have a sense of smell. A most important part is played by this sense in the case of insects which are also frequently originators of odours themselves, for instance for the purpose of defence or also with the object of attracting or recognising other insects or beings. The antennæ are the bearers of this sense in insects. Although some scientists have denied this sense to fishes, there is no doubt of its existence, it being of importance to them in distinguishing food. Apparently, for amphibions animals this sense is not very important, although they also have well-developed organs for this purpose. In the case of reptiles, the value of this sense cannot be underestimated. Some of these animals disengage strong smelling secretions during pairing time or for warding of attacks. In the case of birds, the real smelling

organs are degenerated, for instance it has been proved in reference to such keen-sighted birds as vultures that the sense of smell plays no part in the search of food. For many species of mammalia, the sense of smell is of paramount importance, some of them even being provided with glands in which odoriferous substances are secreted. Bats are more dependant on their sense of touch than on smelling organs, their wing-membranes and the external parts of their ears being especially susceptible. Moles, hedgehogs, martens and some other small mammalia can be designated as smelling animals par excellence. Dogs, of course, must also be mentioned in this category and in point of fact, they seem to have a special sensitiveness for substances which are not easily volatilized as well as for adhesive and putrid odours. Training experiments have proved that they can even distinguish similar smells which are beyond their general experience, for instance nitrobenzene from benzaldehyde.

Henning paid special attention to ants which he was in the habit of observing in their natural surroundings and mode of life in distinction to the majority of other scientists who were wont to study their habits after enclosing them in confined narrow spaces so to speak in "internment camps." His researches were restricted mainly to the habits of the red wood ant *formica rufa,* L. in the forests of Taunus, Spessart, Black Forest and the Vosges.

It goes without saying that such investigations must lead to better and more accurate results when such experiments concerning their smelling sense are performed in the open air than in narrow cages which are quickly permeated by odours. However, one must, in making tests as to sense of smell in ants always pay attention to the fact that their sense of *sight* plays a part thereby. It was already known that ants during their course automatically leave an odoriferous spoor behind them by dropping a secretion on the ground out of their anal glands. The odour of this substance proves it to be formic acid, and Henning's experiments with artificially produced formic acid trails showed that ants during their wanderings are mainly influenced by their sense of smell. However, other odours also, especially such as resemble formic acid in the smelling prism, were apparently suitable as a trail-smell for ants, for instance formic aldehyde and formiates in general. Scents, however, which belong to other groups of the smelling-prism efface the spoor, for instance camphor, benzaldehyde, rosemary and nicotine. If pine needles or other nest-building materials are painted with odoriferous substances, they are either carried to the nest or left alone or removed from the nest, according to the odour of the compound. A single ant is not capable of producing a sufficiently strong-smelling spoor for others to be able to follow it. Several ants are required in order to produce spoors enabling them to find their way home. This is the origin of their state formation. It is an outcome of the structure of their antennæ and is easily understood without any theories concerning social instincts, or special intelligence. The sole explanation is that ants show a strong reaction for formic acid which they produce themselves to which may be added the fact that this compound has a penetrating odour.

Ants recognise eack other neither by sounds nor by rhythms of touch or the sense of sight, but solely by their smell. If an ant is painted with a strange perfume, not belonging to their range of experience, for instance with pineapple oil, it is treated as an enemy by its companions or even killed, and then dragged into the nest as a prey. The painted insect often tries to escape or to purify itself from the strange odour. Animals which have been infected in the same fashion do not fight against each other. Odours which are not far removed from the formic acid range of the odour prism, such as putrid or empyreumatic ones, and also of turpentine and pine needle oils have in diluted concentrations the effect that such insects are avoided by their fellows.

However, no general rule can be laid down in all cases, as ants react also towards complicated odours. All experiments tend to prove that their attitude depends entirely whether the complicated odour they come in contact with is known or unknown to them, whereby such terms as "known or unknown" must not be understood in the sense used in the psychology of the human mind. Ants do not give any proofs of being able to remember facts or of adapting themselves to unexperienced conditions. Ants have quite conformed themselves to the surrounding conditions of their existence; graduated forms of sensitiveness do not appeal to them and their attitude is completely governed by the sum of nervous reactions they are accustomed to; they are quite unable to show a combined reaction towards any two complex stimulations acting simultaneously which would take both into account.

A second appendix treats of the various kinds of taste. In the same manner as a prism is employed as a fundamental model for various odours so in this case a tetrahedron is used for classifying the manifold forms of taste. One imagines the 4 different forms of taste, salt, sweet, sour, and bitter, to be placed at its corners, whereas transitional impressions must be sought for between the corners. The fundamental difference between the psychical impression produced by various tastes in distinction to that excited by various odours consists therein that the former are by no means so easily characterized and defined as the latter.

On the sense of smell and on odoriferous substances in the animal world.—G. Rörig[1]) has continued an article under this denomination of which we already discussed[2]) the first part in our last *Report*. He mentions several cases of the marvellous smelling sense of insects which enables them either to find sources of food or influences the females in finding the right spot to deposit their eggs. For instance an ichneumon-fly *(Rhyssa persuasoria)* bores its eggs into the larvæ of the tailed wasps belonging to the genus *sirex* which live deep down in passages they gradually perforate in living trees whence no trace of their presence is betrayed outside. In spite of this secluded existence, the *Rhyssa* female knows how to find them and her sense of smell is so wonderfully developed that she buries her sting into the right spot of the larvæ-body which is often separated from her by a woodden partition even 3 cm. thick.

Of course errors occur at times which, however, prove absolutely that insects, in depositing their eggs, are guided by their smelling power. Such a case is that of the grey blue-bottle *Sarcophaga carnaria* which places its eggs not only on putrescent bodies of animals and on putrid meat, but frequently also on the leaves of the asclepiadacea *Stapelia hirsuta,* because the latter give off a smell of carrion; of course, in this case the larvæ on creeping out are doomed to perish miserably.

The sense of smell in mammalia plays an important part in enabling the mother-animal to recognise its offspring. Every herd of sheep in which the ewes live together with their lambs affords abundant proof of this fact, as the lambs try to suck all the ewes without any discrimination, whereas the latter only recognise their own offspring and butt off all the rest. Evidently, this discriminating sense of smell is not developed in the lambs, but the old ewes recognise their young however mixed up they are, and in point of fact by their sense of smell, as one can easily convince oneself by testing them. One has also observed in the case of the bear-seals *(Arctocephalus ursinus)* which congregate shortly before the termination of the whelping period in thousands on the Pribiloff Islands that the females are capable of distinguishing their own offspring

[1]) *Deutsche Parf.-Ztg.* 8 (1916), 258, 274. — [2]) *Ibidem* 8 (1916), 214; *Report* Oktober **1916**, 105.

with unfailing accuracy amongst the many thousands that surround them entirely by their sense of smell.

Finally, Rörig discusses the question of the individual smell of animals. The most common odour is probably that of musk. Not only the musk-deer but also the little musk-buck *(Neotragus moschatus)*, a dwarf-antelope of Abyssinian origin whose tear-glands contain the musk-secretion, the musk-ox of Greenland *(Oribus moschatus)*, the American musk-pig *(Dicotyles lapiatus)* whose musk-glands are close to the anus, the North American musk-rat or ondatra *(Fiber zibethicus)*, the musk-shrew *(Myogale pyrenaica)* of Northern Spain, the musk-rat *(Myogale moschatus)* of Southern Europe, and several other species of shrews one and all produce this penetrating scent. To the same category belong the prehensile-foot-jumper of Queensland *((Hypsiprymnodon moschatus)*, a marsupial shrew and the males of the Australian musk-duck *(Biziura lobata)*. Some insects may also be mentioned under this heading, for instance a a bombycid *(Porthesia auriflua)* and the musk-beetle *(Aromia moschata)*.

Observations on the smelling capacity of dogs.—The question if dogs react on odours which affect human beings was hitherto not investigated from an experimental point of view. An essay by C. Heitzenroeder[1]) attempts to solve it.

The external signs of dogs scenting any odour are sniffing movements which become visible when the *nervus olfactorius* is stimulated. These movements can be graphically registered as breathing sensations by means of a girdle attached to the thorax and ending in a so-called Marey's drum. The current of odour is supplied to the dog by means of a hemispherical funnel into which the animal places its snout.

Heitzenroeder was able to demonstrate by this means that dogs react on many odours which also affect human beings. On the other hand, some substance whose odour is quite imperceptible to human beings, owing to their being exceedingly diluted, produce lively sniffing movements in dogs, for instance an extremely diluted solution of female dog's urine.

With the aid of this method, L. Seffrin[2]) tried to determine the keenness of the dog's scent which we referred to in a former *Report*.

Smell and chemical constitution.—We discussed the connection between the constitution and odour of organic compounds in our last *Report* according to a paper by R. Marchand[3]). The same subject has been recently treated by J. V. Dubsky[4]). He also refers to the meagerness of our knowledge of the relations between smell and constitution, discusses the various odorophorous groups, and above all things draws attention to the similarity of odour of substances built up in a completely different manner. Besides, the term "odorophorous groups" one comes across other denominations in literature.

For instance, the term "aromatophorous groups" is sometimes in use, and H. Rupe and K. von Majewski[5]) introduced the designation "osmophorous groups". Even such terms as "kakosmophorous" (evil-smelling) groups, as for instance —SH in the merecaptanes, —S— in the thioethers, —N:C in the carpylamines, are employed.

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[1]) *Über das Verhalten des Hundes gegen einige Riechstoffe;* researches of the Physiological Institute of Giessen. *Zeitschr. f. Biologie* **62** (1913), 491. According to a separate impression kindly placed at our disposal. — [2]) *Über die kleinsten noch wahrnehmbaren Geruchsmengen beim Hund*, researches of the Physiological Institute of the University of Giessen. *Zeitschr. f. Biologie* **65** (1915), 493. (The author kindly sent us a special impression). *Comp. Report* October **1915**, 70. — [3]) *Deutsche Parf.-Ztg.* **1** (1915), 232, 243, 287; *Report* October **1916**, 104. — [4]) *Deutsche Parf.-Ztg.* **2** (1916), 297, 348. — [5]) *Berl. Berichte* **33** (1900), 3401.

Dubsky mentions besides well-known examples of similarity in odour some which are less familiar. He adduces as examples of compounds smelling of oil of bitter almonds not only benzaldehyde and nitrobenzene, but also nitro-thiophene, benzonitrile, azimidobenzene, &c. He even avers that vanilline, *p*-nitroguaiacol and *p*-cyanoguaiacol have the same odour.

The resemblance in smell of bornylacetate and trichlor*iso*butylalcohol (solid acetone chloroform) is said to be absolutely astounding.

He also refers to numerous different compounds which smell of camphor, peppermint, lemons, aniseed, and cinnamon, discusses the influence of the double and threefold linkage, of the methyleneether group and of the methoxyle group on the aroma, and finally that of the individual osmophorous groups.

The chemism of odours is the title of an essay recently published by H. Henning[1]) to which we should like to draw the attention of our readers. He comes to the conclusion — an opinion with which we fully agree — that the chemism of odours is an ambiguous expression because compounds containing quite different osmophores resemble each other in smell, whereas occasionally substances with identical osmophores differ absolutely in scent. However, osmophores must in any case be present, if the compound in question is to have any smell at all.

In spite of all this, he tries to explain the similarity in scent of different compounds by means of a series of diagrammatic formulæ, whereby he classifies the various odours as aromatic, flowery, camphoraceous, resinous, and empyreumatic[2]).

On the influence of volatile oils on the movement of the intestines. — A. L. Muirhead and H. F. Gerald[3]) have investigated the question of the effect of American pennyroyal oil, tansy oil, wormwood oil, turpentine oil, oil of rue, anise oil, savin oil, and of thymol on isolated sections of the intestines. All of these retarded the spontaneous movements of the intestines in concentrations averaging 1:5000 to 1:10000; a few, especially anise oil, oil of turpentine, and wormwood oil, even in concentrations between 1:25000 and 1:50000.

Aromatics as medicines. — O. Anselmino[4]) discusses the importance of aromatics as medicines in a perspicuous form, easily comprehensible even for the uninitiated. The number of distilled waters and alcoholic preparations containing essential oils, but especially the number of the essential oils themselves, mentioned in the official pharmacopœias, has changed. Whereas, *e. g.*, the *Pharmacopœa Borussica ed. IV.* of 1827 contained only 18, the *Pharmacopœa Borussica ed. VII.* of 1862, however, 30 essential oils, and the first German pharmacopœia, the *Pharmacopœa Germanica* of 1872, 32, their number has sunk again to 20 in the 5th ed. of the German pharmacopœia, 1910[5]). The official list of prices of medicines, however, which comprises a wider range of drugs than come into consideration for the requirements of the pharmacopœia, stipulates for the pharmaceutical chemists the maximum prices of about 50 essential oils.

Judging by the number of essential oils in the pharmacopœia, one would think that at least a good many of them had certain peculiar medicinal properties. However, this is not the case to such an extent. Most of them only serve to improve the taste and smell of the medicines, and even this way of using them has diminished.

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[1]) *Deutsche Parf.-Ztg.* **3** (1917), 125. — [2]) *Zeitschr. f. Psychologie* **73** (1915), 161. — [3]) *Journ. Pharm. Therap.* **8** (1916), 253; *Chem. Zentralbl.* **1916**, II. 504. — [4]) *Deutsche Parf.-Ztg.* **3** (1917), 309, 321. — [5]) Comp. *Report* April **1911**, 124.

The preliminary condition for a more extensive use would be, it is true, that the various essential oils differed in action. Their action, however, has not been studied systematically, so far, and reliable pharmacological information is to be found only in isolated cases; ylang-ylang oil, *e. g.*, has been well examined in this respect[1]). According to Anselmino, a systematic investigation of the special effect of each of the various essential oils would not promise any great success, as on a whole many of them are rather closely allied in composition.

The author distinguishes between local and reabsorbing action, which can be brought about by internal administration or inhalation. It can be said in general that all essential oils irritate locally and are poisonous. The quantities given for medicinal purposes or used in order to improve the taste and smell of other substances are so small, however, that no poisonous effect takes place.

Thujone[2]) and myristicin[3]) are looked upon as specifically poisonous, in consequence of reliable investigations. As regards safrole, it is known that, after having been taken internally, it may cause fatty degeneration of the liver and kidneys.

The bad effect that may be caused by ample inhalation of essential oils is sufficiently known; the symptoms, summarized in the term nausea (literally meaning seasickness), are numbness, slight giddiness, headache, &c., such as are observed after excessive use of scents or prolonged stay in rooms where there are strongly smelling flowers. Geranium oil is looked upon as being specially liable to cause nausea.

Mustard oils, generally used in alcoholic solution, are known as extraordinarily strong irritants of the skin. The oils of rosemary, thyme, wild thyme, rue, mace, juniper and geranium also irritate the skin.

The irritative property of essential oils is but little used nowadays in ointments, only the rosemary or nerve ointment still keeps its place, generally speaking. In the veterinary practice and in some popular remedies, turpentine in the form of ointments and plasters still acts a part as irritant, the action of which is chiefly due to its essential oil.

Oil of turpentine and especially fir oils as well as the oil of *Pinus pumilio* have found a modern use in medicinal baths, the action of which is due to irritating properties of the essential oils with regard to the skin[4]).

According to Anselmino, the irritating effect of tincture of arnica on the skin must likewise be ascribed to the essential oil[5]).

Excepting the antiseptic power of some components of the essential oils, the chief aim of their use in dentifrices, tooth-powders, &c. is to modify the taste of these preparations.

Oil of cloves, when applied against toothaches, does not act only as desinfectant but most probably as a local anæsthetic as well. The other remedies against toothache, such as spirits of mustard and oil of cajuput, act through deflexion or diversion. Anselmino thinks it possible that the cheek rubbed with spirits of mustard burns to such an extent that the toothache is forgotten[6]).

The loosening action (oils of turpentine, cypress, eucalyptus and menthol) on the mucous membranes of the nose and the palate and on the respiratory organs consists

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[1]) P. Kettenhofen, inaugural dissertation, Bonn 1906; *Report* April **1907**, 111. — [2]) F. Jürss, *Beiträge zur Wirkung des Thujons* (Contributions regarding the action of thujone). *Bericht* October **1903**, 141. — [3]) F. Jürss, *Über Myristicin und einige ihm nahestehende Substanzen* (On myristicin and some allied substances). *Bericht* April **1904**, 159. — [4]) Comp. O. Anselmino and J. Schilling, *Therap. Monatsh.* **28** (1914), 502. — [5]) There is scarcely anything known of the composition and properties of arnica flower oil, whereas the oil from the roots has been examined fairly well. The tincture, however, is made from the flowers. — [6]) According to Schwarz, it would be sufficient to smell mustard oil in order to make the pain cease! Comp. *Report* April **1914**, 90.

likewise in an irritation, combined with disinfecting and deodorizing power; at the same time the cough is alleviated. Anselmino thinks, the root of the burnet-saxifrage [1]), used as a popular remedy against cough and hoarseness, may owe its efficacity to its content of essential oil.

If essential oils get into the stomach (peppermint lozenges, aromatic liqueurs), the irritation caused by them is noticed as a sensation of heat or slight burning, a further consequence being an acceleration of the digestion and of the discharge of the stomach *(apéritif)*.

The best-known action of essential oils on the intestines is that of removing flatulence, caraway, fennel and anise being specially used for the purpose. Anselmino explains this action by the irritation of the intestines hastening their discharge.

It has not been stated with certainty in what form the oils absorbed by the body are eliminated. They often appear in the urine as unknown compounds, but also unaltered. On their way through the kidneys, they irritate these too, inciting them to increased activity; they act as diuretics and may, in larger quantities, cause inflammation of the kidneys. In any case, it is necessary to watch the kidneys carefully in all treatments with essential oils.

Well-known diuretics are juniper berries (gin), lovage and angelica, furthermore parsley (apiole), the anethol-containing seeds of anise and fennel, and sassafras wood (safrole). Asarone, occurring in the oils of *Asarum europœum,* calamus and matico, is likewise supposed to be a diuretic.

Sandalwood and copaiba oils too act as diuretics. Their antigonorrhœal action, which matico oil has as well, will, however, be due to the fact that they make the urine antiseptic, thus preventing the gonococci from growing.

Whether essential oils are examined in order to determine if those which have in common one peculiar component have the same properties, or, the other way round, if such as are used with the same aim have on the whole the same, or at least a similar, composition, the scanty result will be somewhat disappointing.

The antigonorrhœal remedies, *e.g.,* (excepting perhaps matico oil) contain cadinene as a common component, it is true, but it is surely not the effective one in sandalwood oil, with its about 90 per cent. of santalol, and most likely not the only effective one in the oils of copaiba and cubebs. It would be very natural to try cadinene, which can comparatively easily be obtained in a pure state, as a remedy against gonorrhœa.

The modern medical books indicate the following spheres of use for essential oils:—

For improving taste and smell: oils of bitter almonds, orange flowers, orange peels, bergamot, lemon, lavender, melissa, rose, peppermint, spearmint, cinnamon, fennel, aniseed and sassafras.

For embrocations: oils of arnica [2]), calamus, mace, rue, juniper, savin, rosemary, thyme, wild thyme, mustard, amber, sweet birch and gaultheria (wintergreen).

Against toothache: oils of cloves, cajuput, cinnamon, amber, chamomile and Roman chamomile.

To aid digestion: oils of wormwood, chamomile, Roman chamomile, dill, caraway, coriander, aniseed, fennel, calamus, cloves, cinnamon, cajuput, peppermint, spearmint and sage.

To remove flatulence: oils of chamomile, Roman chamomile, aniseed, star-anise, caraway, fennel, mace, calamus and juniper.

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[1]) We know next to nothing about the properties and constitution of the oil of the burnet-saxifrage roots. — [2]) Arnica flowers contain about 0.55 per cent. of oil.

As expectorants: oils of aniseed, star-anise, sage, eucalyptus, burnet-saxifrage, turpentine and cypress.

As stimulants: oils of cajuput, rosemary and valerian.

As nervine: oil of valerian.

As anthelminthics: oils of cajuput, semencine, wormseed *(chenopodium)*, male fern, eucalyptus and laurel.

As diuretics: oils of angelica, lovage, parsley, sassafras, turpentine, copaiba and sandalwood.

Against gonorrhœa: oils of copaiba, sandalwood, cubebs and matico.

Against nocturnal perspiration: oil of sage.

As sudorific: oil of sassafras.

As antispasmodic: oil of melissa.

Against bleeding of the womb: oil of cinnamon.

Against itch: oil of rosemary.

As to the other aromatics in pharmacy, apart from the plants yielding essential oils, there are resins, balms, animal drugs, and some isolated and artificial aromatics.

Benzoin, myrrh and olibanum are used no longer for fuming the sick-rooms, but benzoin and myrrh, mostly in the form of tinctures, are used in dentifrices and cosmetic lotions.

Balsam of Peru is still frequently used for different purposes; balsam of Tolu, in a lesser degree.

Musk was formerly in repute as a powerful excitant, in many cases the last remedy given to dying people, in order to maintain or bring back consciousness for a short time. Experimental pharmacological investigations, however, have not confirmed such an action.

As to synthetics, and aromatics isolated from essential oils, the following are employed: methyl salicylate (as artificial wintergreen oil), benzyl benzoate (as artificial balsam of Peru), coumarin, heliotropin and vanillin, in order to improve odour and taste, menthol as lenitive, anæsthetic and antiseptic in dentifrices, as menthol crayons, as ointment against cold, itching of the skin, &c. Thymol is used as an antiseptic and against worms.

Anselmino considers it to be doubtful whether fresh spheres of use may be found for aromatics through systematic investigation.

Volatile compounds of urine. — Some time ago, we reported about a volatile compound of human urine to which W. M. Dehn and F. A. Hartman[1]) applied the term urinod and declared it to be probably identical with 3-*cyclo*hexenone-1. Their supposition seems to have been erroneous, as R. J. Anderson[2]) who has also investigated volatile urine compounds believes that he has demonstrated that urinod is a mixture of a neutral substance ($C_7H_{12}O$?) with *p*-cresol.

From cow's urine, he isolated by means of distillation with sulphuric acid a compound $C_{10}H_{16}O$, isomeric to camphor and boiling at 102° (1 mm). When heated it reduces a solution of silver in ammonia, decolorizes a solution of permanganate and absorbes bromine, when dissolved in chloroform, thereby generating hydrobromic acid. The semicarbazone melts at 165°. The compound $C_{10}H_{16}O$ could only be obtained in a pure state from summer urine, but not from winter urine. The ketone also occurs

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[1]) *Journ. Americ. chem. Soc.* **36** (1914), 2118, 2136; *Report* October **1915**, 72. — [2]) *J. Biol. Chem.* **26** (1916), 387, 401, 409; *Journ. chem. Soc.* **110** (1916), I. 773, 774.

in goat's urine, but is not to be found in either horse's or human urine, in which it seems to be replaced by a similar compound, the above-mentioned neutral substance $C_7H_{12}O$ (?).

Physiological effect of d-, l- *and* i-*camphor.* — The question as to whether *d*-, *l*- and *i*-camphor are to be regarded as equivalent from a pharmacological point of view has — apart from its theoretical value — become important because owing to the war the importation of *d*-camphor (Japanese camphor) is excluded, and hence pharmacologists and medical men have to decide if Japanese camphor can be substituted by the artificial inactive camphor.

The Prussian scientific committee for medical concerns *(Preuß. Wissenschaftliche Deputation für das Medizinalwesen)* answered this question in their verdict of October 7th, 1914 to the effect that it would be a risky matter to substitute[1]) *d*-camphor at least for subcutaneous and internal applications by the artificial product with equanimity.

According to G. Joachimoglu[2]), this committee arrived at this result since exact clinical observations on the effects of artificial camphor were wanting, as on investigating both *d*-, *l*- and *i*-camphor he found that practically in reference to their toxicity and to the symptoms of disease they produce there was no difference whatever between them. He determined the letal quantity of the three camphor preparations required by making intraperitoneal injections of their oily solutions into cats. The effects of *d*-camphor *(Pharm. Germ.)* differed in no manner whatever from those of *l*-camphor, m. p. 176 to 177°; $[\alpha]_D$ — 40°, supplied by us, and to which a greater efficacy is frequently attributed. Besides, *i*-camphor, the artificial product manufactured from oil of turpentine, which nowadays has acquired so much importance as a substitute owing to the natural product not being obtainable, showed no difference whatever in comparison to both its optically active compounds, as long as a pure compound (Schering, m. p. 172°; $[\alpha]_D$ + 3,025°) was employed. A preparation of Boehringer (m. p. 172°; $[\alpha]_D$ + 0.225°) which apparently contained impurities and gave a weak chlorine reaction produced a letal effect only when a dose $1^1/_2$ times as strong as the other compounds was employed. Neither in reference to their action on the central nervous system (acceleration of breathing, convulsions) could any difference between the 3 isomeric camphors be observed.

Whereas these experiments were undertaken mainly for practical purposes in order to compare the efficacy of the 3 isomeric camphors, P. Leyden and R. von der Velden[3]) have investigated in what manner a considerable number of camphor preparations act on the heart. They examined to what extent these substances which were dropped directly on to the exposed frog's heart were capable of neutralising the reduction in frequency produced on it by chloralhydrate. In this case also *l*-camphor proved to be about on a par with the *d*-product. On the contrary, the artificial *i*-camphor as well as a mixture of both the optical isomerides produced no action whatever on this experimental material. Slight alterations in the form ot the camphor molecule (I), as they are caused in epicamphor (*β*-camphor) (II)[4]) by the greater distance between the methyl and ketone group, in ethylcamphor (III) by the substitution of the methyl by the ethyl group, in camphenilone (IV) by the addition of a second methyl group, quite obliterate any effect on the heart. In like manner the pure hydrocarbon, camphene (V)

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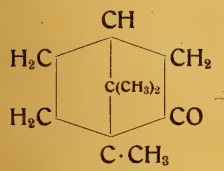
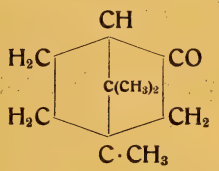
[1]) Comp. *Report* April **1915**, 76. — [2]) *Arch. f. experiment. Pathol. u. Pharmakol.* **80** (1916), 1; *Apotheker Ztg.* **31** (1916), 393. — *Therap. Monatsh.* **30** (1916), 450. — *Chem. Zentralbl.* **1916**, II. 408. — *Berichte d. deutsch pharm. Ges.* **26** (1916), 427. — [3]) *Arch. f. experiment. Pathol. u. Pharmakol.* **80** (1916), 24; *Therap. Monatsh* **30** (1916), 450. — *Chem. Zentralbl.* **1916**, II. 408. — [4]) Comp. *Report* April **1914**, 141.

in which the ketone group is missing is just as ineffective as thujone (VI) which only differs from camphor owing to an alteration in the bridge formation, whereas both these compounds by altering the experimental method (action of their vapour on the frog's heart) showed themselves to be efficacious to former experimentalists[1]). These investigations also demonstrated in this modified experimental method the uniform efficacy of *d*- and *l*-camphor and the total ineffectiveness of *i*-camphor. Comparative experiments on the action on the heart, when using various solvents for camphor lead to the conclusion that every other solvent rather than oil makes the effect of camphor more plainly visible.

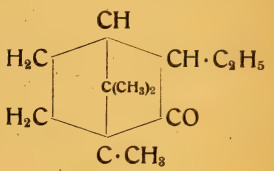
In reference to these theoretical reflections we should like to make the following remarks:—Firstly, in ethylcamphor no methyl group is substituted by an ethyl group, but rather a hydrogen atom of the methylene group next to the carbonyl one is substituted by ethyl. Camphor in which the methyl linked to the nucleus is substituted by ethyl would be an *1*-ethyl-*7,7*-dimethylbi*cyclo*(*1,2,2*)-heptanone, a compound which is still unknown. Besides, camphenilone does not contain one methyl group *more*



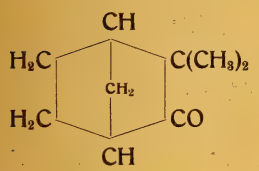
(I) Camphor.



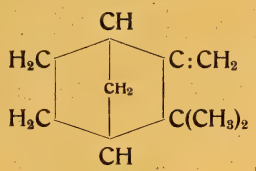
(II) Epicamphor.



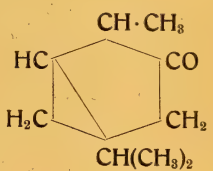
(III) Ethylcamphor.



(IV) Camphenilone.



(V) Camphene.



(VI) Thujone.

than campher, but rather one *less*. Finally, thujone is not distinguished from camphor by a modification of the bridge formation, on the contrary it is a compound having an utterly different constitution.

In like manner experiments on isolated frog's hearts with the 3 camphor varieties left no doubt as to the similarity of their effects from a pharmacological point of view. Nor was Joachimoglu with the aid of experiments able to detect any difference in their effects on bacteria.

Joachimoglu has made experiments concerning the heart action of camphor on isolated frog's hearts according to Straub's method, employing for this purpose solely watery camphor solutions. With this object in view he determined the solubility of camphor in water and found that one part of *d*-camphor dissolves in 666 parts of water, whereas one part of *l*- or *i*-camphor requires 800 parts of water in order to dissolve. For the purpose of testing the antiseptical effects of camphor the *Bacterium coli* and the *Vibrio Metschnikoff* were chosen. The camphor was dissolved in sterilised nutrient broth and this camphor-broth was diluted accordingly by means of normal broth. After this procedure the tubes were inoculated with a broth-culture prepared

[1]) Comp. W. Heubner, *Therap. Monatsh.* **27** (1913), 593; *Report* October **1913**, 127.

24 hrs. beforehand. The tubes remained for a week in the incubator at 37° and were examined every day in reference to the development of the bacteria. As already mentioned above, no difference in their antiseptical effects was perceptible between the 3 camphor varieties.

The theoretical importance of both these investigations is to be found in the demonstration of the equivalency of *d*- and *l*-camphor. In reference to the artificial inactive camphor there is only room for some uncertainty in so far as it apparently shares with natural camphor equally its stimulating effects on the central nervous system, whereas it does not seem to be of any use as a substitute for exciting the heart's action. From the point of view of the synthesis of medical preparations, von Leyden and von der Velden have undoubtedly demonstrated the important fact that no chemical modification of camphor can be employed as a substitute in the treatment of the heart.

The abstractor of the *Therapeutischen Monatshefte* remarks on this matter that it is still an open question how far the above statements apply to borneol which the authors did not examine: anyhow, there is no doubt that von Leyden's and von der Velden's thesis does not apply to norcamphor[1]) which, according to the abstractor's opinion seems to approach as nearly as possible the solution of another important problem, that of a camphor easily soluble in water, a process for preparing solutions of norcamphor fit for injections.

The non-appearance of a colouring with vanillin and hydrochloric acid during 24 hours[2]) served as a criterion of the purity of the camphor preparations. As a so far-reaching purification could not be attained by means of recrystallisation, but solely by the help of the osazones, and in this case the slight rotation which commercial samples of artificial camphor show disappears completely.

In addition to the above-mentioned researches, G. Leone[3]) has studied the pharmacodynamical effects of camphor on the main arterial system and on the isolated heart. The effects on the main arterial system consist, as Liebmann[4]) indicates for the smaller one, in a reduction of the arterial pressure and in an increase of the amplitude of the heart beats, when small and medium doses are given (0.25 to 1 cg. per kg.) which is occasionally preannounced or followed by a short increase of the number of beats, during which clonical twitching of the limbs is perceptible; when large doses, sufficient to produce convulsions (2 to 5 cg.) are given the pressure is increased thereby, after a slight, temporary reduction. Smaller and medium doses further produce enlargement of the blood vessels. An increase in the amplitude of the heart beats is also perceptible after large doses and must be attributed to the exciting effect exerted on the excitomotorical apparatus of the heart. Camphor increases the number and amplitude of the heart beats when acting on isolated hearts and brings them, even after they have been almost totally silenced by poisoning with chloralhydrate, back again to their normal function.

It is not apparent from the abstract what kind of camphor was used for these experiments; however, one must assume that it was the ordinary *d*-, Japanese camphor.

Camphorated wine for treating wounds.— R. Koch[5]) obtained very good results in treating infected wounds with camphorated wine instead of with acetate of alumina.

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[1]) Bayer & Co., D. R. P. 289950; *Therap. Monatsh.* **30** (1916), 342. — [2]) Comp. *Report* April **1915**, 77; *Bericht* October **1916**, 60. — [3]) *Arch. d. Farmacol. sperim.* **21** (1916), 370; *Chem. Zentralbl.* **1916**, II. 503. — [4]) *Chem. Zentralbl.* **1912**, II. 46; *Report* October **1912**, 138. — [5]) *Therap. Monatsh.* **29** (1915), 175, 251.

He employed the camphorated wine according to the prescription of the *Pharm. Germ.* ed. V.: —

Rp. camphor . . . . . 1 part
alcohol . . . . . 1 „

which are gradually mixed with

mucilage of acacia . . . . . 3 parts
white wine . . . . . 45 „

To be shaken before using.

In this form camphor is apparently a strikingly good vulnerary. The edges of the wounds do not look pale and inflated, as is usually the case when moist bandages are employed; on the contrary, when it is a question of vascularized skin, they have a normal red colour, the granulations are large and fresh, the purification of the wound takes place in a strikingly short space of time, and the anodynous effect is greatly appreciated by the patients. Koch employed camphorated wine in all cases in which usually moist bandages are used, accordingly for the treatment of smeary, ill-healing, painful, irritated wounds. Camphorated wine macerates and inflates the skin less than acetate of alumine.

Koch was hitherto unable to decide the question if camphor wine accelerates the healing of such wounds as tend to heal very slowly. However, there is no doubt that the conditions of circulation in the neighbourhood of the wounds by no means deteriorate, but often vastly improve, that it excites the formation of granulations, reduces the secretion of pus, and allays pain.

It further has the advantage over pure alcohol bandages that its use is not painful. Koch also tried to make dry bandages filled with pure finely divided camphor, but without any certain results.

The effects of this treatment often diminish, in which case it has been found to be advisable to vary them with dry bandages.

On the *sterilization of catgut by means of eucalyptole or juniperwood oil,* see page 91 of this *Report.*

Menthol-eucalyptole for the treatment of diseases of the respiratory channels. — The treatment of tuberculosis by means of menthol-eucalyptole recommanded by Berliner [1]) has, according to Rotky [2]) only then a probalility of success, when the disease has not made too great progress, especially in such cases in which both lobes of the lungs have not been infected. Especially when high fever prevails, a favourable effect of the treatment appears doubtful. If only a part of the lung tissues is diseased, so that only the catarrh is the typical manifestation of it, and if the state of nutrition is satisfactory, it is quite possible to arrive at good results by means of a protracted menthol-eucalyptole treatment. It is especially well worth while attempting this method with such patients who for any not defined reasons are prevented from undergoing climatic cures.

The object to be aimed at is to bring about the total disappearence of the tendency to cough which originates in the uppermost parts of the breathing passages, as for instance in the larynx and in the upper pharynx, by means of the anæsthesis of these parts. This purpose is said to be best attained by a combination of anæsthesine and

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[1]) Comp. *Report* April **1915**, 77. — [2]) *Prager mediz. Wochenschr.* **1914**, 523; *Zentralbl. f. innere Medizin* **1915**, 729. After Merck's *Jahresbericht* **29** (1915), 327.

of other suitable remedies which have an anæsthetic effect on the mucous membrane of the mouth and larynx. It runs as follows: —

Anæsthesine	0.03
Phenacetine	0.08
Thymol	
Menthol	
Ol. Eucalypti *ana*	0.0015

and is termed *Thyangol.*

Six to ten tablets are taken daily, and the patient's object is to facilitate the dissolution of the medicine at the back part of the tongue, in order also to moisten the mucous membrane of the larynx with the saliva containing the remedy.

According to Gutstein[1]), these tablets have been a decided success in practice, even in cases in which the lower respiratory organs were affected, as for instance in bronchitis and in tuberculosis of the lungs. They are also said to be particularly useful for the treatment of tuberculosis of the larynx.

On the other hand, F. Bode[2]) experienced most satisfactory results by eucalyptole menthol injections in cases of bronchitis and pneumonia which had been operated. This treatment was recommended by Technau[3]); the prescription he employed originally advocated by Berliner is the following: —

Menthol	10 g.
Eucalyptol albiss. (Schimmel)	20 g.
Ol. Dericini	50 g.

One ccm. to be injected daily intramuscularily.

Bode was accustomed to make injections of eucalyptole-menthol even a few days before operating on old people and such patients as suffered from existing bronchitis or a tendency to lung affections; this mostly had the satisfactory effect of preventing ulterior complications. It is of importance to continue the treatment for some time in order to obtain satisfactory results. In cases of feverish bronchitis and of broncho-pneumonia, the temperature diminishes lytically in quite a short time, the cough becomes looser, abundant expectoration supervenes and then disappears again totally after a few days. Also in the after-treatment of tracheotomized diphteria-diseased children these injections proved to be most beneficial. In distinction to adults, relatively smaller quantities (about half those mentioned above) were injected; irritation of the kidneys was never observed.

Methylheptenone as an insecticide. — The tsetse fly which is so much feared as a transmitter of sleeping-sickness is said to have a decided aversion to citronella grass and lemongrass[4]).

Probably the effect of the latter depends on its methylheptenone content[5]). Experiments have been conducted in this direction with the result that methylheptenone is now employed as an insecticide, especially in the French army.

In point of fact, lemongrass oil does not contain any large quantity of methylheptenone, however it can be obtained in larger quantities by the action of alkalies on citral.

Methylheptenone is said to be much more efficacious than anisole for this purpose.

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[1]) *Medizin. Klinik* **1915**, 1295. — [2]) *Münch. med. Wochenschr.* **1916**, N° 9; *Allg. med. Zentral-Ztg.* **86** (1917), N° 8, p. 30. — [3]) Bruns' *Beiträge z. klin. Chirurgie*, vol. 91. — [4]) Vide *Report* October **1909**, 44; April **1910**, 75. — [5]) *Perfum. Record* **7** (1916), 295.

Physiological effects of d-*pinene hydrochloride and of* d-*camphene on frogs.* — S. Dontas and D. E. Tsakalotos[1]) have investigated the effects of *d*-pinene hydrochloride and of *d*-camphene on the heart and respiration of frogs in comparison to those of *d*-camphor and of *d*-pinene. They have ascertained thereby that the physiological effects of *d*-camphene bear the strongest resemblance to those of *d*-pinene, whereas those of *d*-pinene hydrochloride can hardly be distinguished from those of *d*-camphor. The only difference in the attitude of the two last-mentioned compounds consists therein that *d*-pinene hydrochloride produces an even stronger palpitation of the heart than *d*-camphor, but its efficacy is of shorter duration. Besides this, *d*-pinene hydrochloride does not affect the respiration in the least, whereas *d*-camphor exerts a very strong influence in this direction.

On the diuretic effect of an infusion of spike blossoms, vide page 60.

On the hæmolytic effect of thymol, vide page 101.

On the use of thymol as a mouth wash, vide page 101.

On the physiological effect of wormseed oil, vide page 80.

On the treatment of itch by means of cinnamaldehyde, vide page 90.

Chemical Notes.

The use of heliotropin, cinnamaldehyde, and of methyleugenol etc. as raw material for the production of compounds of importance from a therapeutic standpoint. — The researches on the constitution of natural alkaloids which are so important in therapy have proved that many of them are derivates of quinoline or *iso*quinoline. Accordingly, the question of employing quinoline or *iso*quinoline for building up medical compounds was of special interest, and as a matter of fact gradually numerous derivatives of these bases have been produced which are in reality efficacious remedies in therapy or at least said to be so. The effect, namely, of many of these preparations, generally manufactured according to patented processes, is still very incompletely known; others amongst them have not succeeded in practice and only a few of them have hitherto been able to assert themselves. Even the synthesis of quinine, the most important alkaloid, has not succeeded yet in spite of the fact that the consumption of quinine is very much greater than that of all other artificial remedies.

The reason why we wish to discuss the building up of some derivatives of quinoline or other compounds related to alkaloids is simply because many of them are derived from preparations which we manufacture ourselves. For this purpose we quote a series of essays by G. Cohn[2]), entitled, "Artificial remedies of the quinoline and *iso*quinoline group."

For instance, in order to produce *2*-piperonyl-quinoline-*4*-carboxylic acid, heliotropin (piperonal) is boiled according to a patented process with aniline and pyro-racemic acid in alcoholic solution[3]).

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[1]) *Journ. de Pharm. et Chim.* VII. **15** (1917), 12; *Apotheker Ztg.* **32** (1917), 108. — [2]) *Pharm. Zentralbl.* **57** (1916), 663, 704, 715, 732, 749, 768, 788, 807, 826; **58** (1917), 70, 83, 95, 107. — [3]) *D. R. P.* 244497; *Chem. Zentralbl.* **1912**, I. 690.

By boiling *p*-toluidine with heliotropin and pyro-racemic acid, *2*-piperonyl-*6*-methylquinoline-*4*-carboxylic acid is produced[1]).

Further heliotropin, when boiled in alcoholic solution with *p*-aminophenol and pyroracemic acid, produces *2*-piperonyl-*6*-hydroxyquinoline-*4*-carboxylic acid which is then purified over its sodium salt[2]).

By condensing heliotropin with *o*-anisidine and pyroracemic acid *2*-piperonyl-*8*-methoxyquinoline-*4*-carboxylic acid is produced which can than be purified by means of repeated precipitation from its alkaline solution[3]). All these acids are remedies for gout and rheumatism in the joints, the *2*-phenylquinoline-*4*-carboxylic acid (phenylcinchoninic acid) has, however, the disadvantage of possessing a rather bitter taste. Piperonyl cinchoninic acid, on the contrary, is far more palatable, although its therapeutic effects are just as satisfactory. Salicylic aldehyde is employed for the producing of *6,6'*-diquinolyl-*2,2'*-dihydroxyphenyl-*4,4'*-dicarboxylic acid and in point of fact by condensating it with pyroracemic acid and benzidine[4]).

By boiling *p*-aminoacetaldehyde, alcohol, pyroracemic acid and anisic aldehyde, one obtains the acetylated *2*-*p*-methoxyphenyl-*6*-aminoquinoline-*4*-carboxylic acid which produces the free acid on saponification; as a derivate of quinoline it is of importance from a therapeutic standpoint and is said to exercise a similar effect to atophan[5]).

Heliotropin which is obtained from safrole[6]) is also remarkable as a raw material for the production of *iso*quinoline compounds amongst which hydrastinine plays an important part.

The first original synthesis of hydrastinine was performed by P. Fritsch[7]). He coupled piperonal (heliotropin) with aminoacetale, forming piperonal aminoacetale, and obtained from the latter by closing the ring *6,7*-methylenedihydroxy*iso*quinoline which he transformed into hydrohydrastinine by means of methylation and subsequent reduction. Fritsch's synthesis, however, gave no good yields in practice and has accordingly been abandoned for technical purposes.

All later syntheses whose elaboration has been mainly the merit of H. Decker and A. Pictet start from homopiperonylamine which is produced according to various methods that, however, one and all have to employ heliotropin. —

If one allows formaldehyde to react with homopiperonylamine, so-called Schiff base is formed, namely methylenehomopiperonylamine which can be readjusted to produce the isomeric norhydrohydrastinine. As, however, the aldehyde is a strong methylating agent the reaction between formaldehyde and homopiperonylamine can be so conducted as to form hydrohydrastinine directly. One can also arrive at the same result by letting methylhomopiperonylamine act on formaldehyde[8]). The product of the reaction of formaldehyde on homopiperonylamine is formylhomopiperonylamine which is transformed by internal coupling into *6,7*-methylendihydroxy-*3,4*-dihydro-*iso*quinoline (norhydrastinine) and the latter by means of methylation turns into the hydrastinine-salt. Inversely, one can first methylate the homopiperonylamine, then obtain the formyl compound from the methylhomopiperonylamine, and finally couple the latter directly to hydrastinine[9]).

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[1]) *D. R. P.* 281603; *Chem. Zentralbl.* **1915**, I. 232. — [2]) *D. R. P.* 246078; *Chem. Zentralbl.* **1912**, II. 1677. — [3]) *D. R. P.* 281603; *Chem. Zentralbl.* **1915**, I. 232. — [4]) *D. R. P.* 246078; *Chem. Zentralbl.* **1912**, II. 1677. — [5]) *D. R. P.* 294159; *Chem. Zentralbl.* **1916**, II. 707. — [6]) Cohn states that safrole is the main component of caraway oil; he probably meant carvone, as caraway oil does not contain any safrole. — [7]) *D. R. P.* 86561; *Chem. Zentralbl.* **1896**, II. 224. — [8]) *D. R. P.* 257138; *Chem. Zentralbl.* **1913**, I. 1154. — H. Decker and P. Becker, *Liebigs Annalen* **395** (1913), 342, 351. — [9]) *D. R. P.* 234850; *Chem. Zentralbl.* **1911**, II. 112; *D. R. P.* 245095; *Chem. Zentralbl.* **1912**, I. 1267; *Liebigs Annalen* **395** (1913), 382.

The action of homopiperonylic acid on homopiperonylamine produces homopiperonoylhomopiperonoylamine. The coupling of this amide to an *iso*-quinolinederivate shapes itself just in the same manner as with formylhomopiperonylamine, whereby piperonylnorhydrastinin results from which one can obtain by methylation and subsequent reduction a tetrahydro-*iso*-quinoline derivate, the latter forming hydrastinine and piperonyl on being oxidised, which can be employed again for a repetition of the operation.

Homopiperonoylhomopiperonylamine can also be manufactured from piperonal and hippuric acid, whereby the formation of homopiperonylamine is avoided[1]).

Homologous products of hydrastinine can also be prepared with the aid of piperonal derivates[2]).

For instance by treating benzenehomopiperonylamine with fairly strong hydrochloric acid the hydrochloride of *1*-phenyl*nor*hydrohydrastinine is formed. One can obtain this compound also by boiling the benzene derivative with benzene and phosphorus oxychloride, and equally so by reduction with phenylnorhydrastinine.

For the purpose of preparing *1*-methyl*nor*hydrohydrastinine, homopiperonylamine is heated on the water-bath with acetic aldehyde solution and the resulting compound, dissolved in benzene, treated with hydrochloric acid gas.

Cinnamaldehyde and homopiperonylamine react in producing cinnamylidene norhydrohydrastinine; cinnamylidenehomopiperonylamine which is formed thereby is rearranged in the same manner as the above-mentioned acetic aldehyde compound.

1-Methylenedihydroxyphenylnorhydrohydrastinine is an oil which can also be obtained in crystals. For this purpose, piperonal and piperonylamine in alcohol are heated and one then rearranges the resultant Schiff base by heating it with phosphorus oxychloride in a solution of toluene.

Homologous hydrastinines which are combined with other alkyls instead of with methyl at the nitrogen atom can be obtained by adding alkylhaloids to norhydrastinine[3]), identical or analogous compounds by means of internal coupling of alkylacylhomopiperonylamines. The position *1* of these hydrastinines is substituted if, instead of starting from formyl-, one proceeds from other acylhomopiperonylamines. In this manner one obtains bases which produce vascular distensions just like hydrastinine[4]).

Thus *n*-ethylnorhydrastinine can be produced by preparing ethylhomopiperonylamine *via* the benzoate, then by heating the former with the calculated quantity of formic acid, and by coupling the formyl compound to the *iso*quinoline derivate by boiling the latter in a solution of benzene with phosphorus oxychloride.

For the purpose of manufacturing *1*-phenylhydrastinine, methylhomopiperonylamine is heated with benzoic anhydride and the resulting benzoyl compound boiled in the usual manner with toluene and phosphorus oxychloride. Or one can also transform homopiperonylamine by the aid of benzoylchloride into the benzoyl compound and boil the latter with toluene and phosphorus oxychloride, whereby *1*-phenylnorhydrastinine is formed, combines with methyl iodide, forming *1*-phenylhydrastinine iodide.

1-Methylhydrastinine can in like manner be obtained from homopiperonylamine by boiling its acetate, dissolved in toluene, with phosphorus pentoxide. The *1*-methylnorhydrastinine thus produced is transformed into *1*-methylhydrastinine by means of methyl iodide.

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[1]) W. Kropp, H. Decker and C. Zoellner; *Berl. Berichte* **42** (1909), 1184. — [2]) H. Decker and P. Becker, *Liebigs Annalen* **395** (1913), 342; *D. R. P.* 257138; *Chem. Zentralbl.* **1912**, I. 1154. — [3]) H. Decker and P. Becker, *Liebigs Annalen* **395** (1913), 325. — *D. R. P.* 267699; *Chem. Zentralbl.* **1914**, I. 201. — [4]) H. Decker, *Liebigs Annalen* **395** (1913) 282. — *D. R. P.* 235358; *Chem. Zentralbl.* **1911**, II. 171.

With the object of producing *1*-benzylhydrastinine, homopiperonylamine is heated with phenylacetic acid, the resultant phenylacylhomopiperonylamine boiled with toluene and phosphorus pentoxide, and the *1*-benzylnorhydrastinine thus obtained finally treated with methyl iodide.

3-Alkylhydrastinines are prepared by first combining *C*-alkyl-*N*-(aralkyl)homopiperonylamines with formic acid and then treating them with an acid coupling medium[1]). In order to produce *3*-methylhydrastinine, one starts thereby from methylenedihydroxyphenyl-*N*-methylformyl*iso*propylamine; *3*-methyl-*N*-ethylnorhydrastinine is prepared from *3*-methyl-*N*-formyl*iso*propylamine by heating it with phosphorus pentoxyde in toluene solution and treating the resulting *3*-methylnorhydrastinine with ethyl iodide.

Cotarnine is another important alkaloid; a number of its salts (styptol, stypticin) have been introduced into medical circles as hemastatics, but it is not so useful as hydrastinine from which it differs by having one methoxyl-group more. Accordingly the syntheses of hydrastinin can be adapted with the necessary variation also for the manufacture of kotarnin.

Two general methods are known for the synthesis of cotarnine, one having been discovered by H. Decker[2]) and the other by A. H. Salway[3]).

In both cases the raw material is myristicine which is transformed *via* myristicinaldehyde into methoxyhomopiperonylamine. Decker obtains cotarnine from the above-mentioned base according to a method analogous to that of the preparation of hydrastinine, whereas Salway proceeds in a different fashion.

Decker lets formylhomomyristicylamine react with phosphorus oxychloride in a solution of toluene and works up the resultant norcotarnine as usual.

Salway treats phenylacylhomomyristicylamine with phosphorus pentoxide; thereby a mixture of two isomeric bases is formed which are separated from each other by means of fractionated crystallization of their hydrochlorides. One of them is *1*-benzylnorcotarnine, the other *1*-benzylneocotarnine. The former is transformed into the methylchloride which produces *1*-benzylhydrocotarnine by the agency of reducing substances, whereupon oxidation leads to cotarnine, the benzyl group being split off. Hydrocotarnines which are substituted in the position *1* can be prepared like the corresponding hydrohydrastinines by the action of organic magnesium compounds on cotarnine. For practical purposes, the chloride of the base and an excess of Grignard's solution is employed in order to obtain a good yield.

The synthesis of berberine starts from homopiperonylamine which one couples with homoveratric acid chloride and thus obtains homoveratroylhomopiperonylamine. This amide is then condensed in the usual manner into a dihydro*iso*quinoline derivative, the latter reduced by the aid of tin and hydrochloric acid to a tetrahydro*iso*quinoline derivative which finally reacts with methylal producing tetrahydroberberine. The raw material for the manufacture of veratric acid is either eugenol or vanillin. Berberine is prepared from tetrahydroberberine as usual by oxidation.

Finally, only the synthesis of laudanosin remains to be discussed for which purpose veratric aldehyde (methyl vanillin) is used, which, when coupled with acetic anhydride and sodium acetate leads to dimethoxycinnamic acid, the latter producing dimethoxyhydrocinnamic acid by the aid of reducing agents. This acid is then transformed with the help of its amide into homoveratrylamine[4]).

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[1]) *D. R. P.* 279194; *Chem. Zentralbl.* **1914**, II. 1174. — [2]) *D. R. P.* 245095; *Chem. Zentralbl.* **1912**, I. 1267. — *Liebigs Annalen* **395** (1913), 328. — [3]) *Journ. chem. Soc.* **97** (1910), 1208. Comp. also F. L. Pyman, *ibidem* **95** (1909), 1738. — [4]) A. Pictet and M. Finkelstein, *Compt. rend.* **148** (1908), 925. — *Berl. Berichte* **42** (1909), 1979.

The same result is arrived at by starting from homoveratric aldehyde (obtained by oxidation of methyleugenol), converting it into its oxime, and reducing the latter.

Homoveratrylamine is combined with homoveratric acid chloride, producing an amide which gives in the usual manner a dihydro*iso*quinoline derivative, *i. e.* dihydropapaverin. On reducing the latter one obtains a tetrahydro compound, *i. e. d,l*-laudanosin which can be split up into its optically active components.

Hydrocarbons.

The melting point of doubly unsaturated bodies with conjugated double bonds. — The melting point of unsaturated acids which posess a double bond next to the carbonyl group are a good deal higher than those of the corresponding saturated compounds. Accordingly, heptatriene which has been recently prepared by C. J. Enklaar[1]) has a much higher melting point than heptane. Enklaar[2]) also determined the melting point of a series of other hydrocarbons, but a comparison with the corresponding saturated hydrocarbons was not possible. The figures he found were the following: —

	congelation point:	melting point:
Isoprene	about — 120°	not sharply defined
β,γ-dimethylbutadiene .	below — 65°	— 65 to — 55°
α,α-dimethylbutadiene .	about — 70°	not scharply defined
hexadiene	66°	not scharply defined
dihydromyrcene . . .	at low temperatures it becomes first sirupy and then glass like	
dimethyl-2,6-octane . .	at low temperatures it becomes first sirupy and then glass like	
octatriene	—	— 90 to — 85°
nonatriene	does not become solid even at — 70°	

Investigations on ocimene and analogous hydrocarbons. — We have frequently had occasion to discuss C. J. Enklaar's[3]) publications on ocimene and similar hydrocarbons and can now again report on researches of the same nature by this author[4]).

By ozonizing allo-ocimen[5]) with the aid of ozone of 5 to 8 per cent. O_3-content, he obtained an ozonide which was almost completely soluble in water. The watery solution contained traces of a substance which formed with phenylhydrazine a compound melting at 230°. Apparently this substance is only obtained in the presence of ozone of a high percentage and it is not a normal product of disintegration. This oxidation certainly does not lead to trioxymethylene. Dihydro*allo*ocimene was also ozonized; the watery solution of the decomposition-product only gave a slight pyrrole reaction; acetone could not be detected, however, with certainty an aldehyde or ketone boiling at about 80°. Nor had levulinic acid been formed.

The watery solution of the ocimene ozonide which contained the non-volatile products gave with zinc dust a strong pyrrole reaction.

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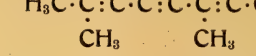
[1]) *Berl. Berichte* **49** (1916), 211; *Report* October **1916**, p. 117. — [2]) *Recueil trav. chim. des P.-B.* **36** (1916), 247. According to a separate impression kindly placed at our disposal. — [3]) Comp. *Report* April **1906**, 109; October **1908**, 153, April **1909**, 119. — [4]) *Recueil trav. chim. des P.-B.* **36** (1916), 215. According to a separate impression kindly placed at our disposal. — [5]) *Allo*ocimene originates from ocimene hy applying heat.

Enklaar assumes on the strength of the results of these and former investigations the following formula for *allo*ocimene: —



However, he is not inclined to regard this formula as final till larger quantities of the ozonide have been examined.

On this basis ocimene would apparently have the following formula: —

It is just possible also that ocimene and *allo*ocimene are mixtures like the citronellals. The formula for *allo*ocimene which Enklaar suggests is the same Auwers and Eisenlohr[1]) formerly proposed.

Enklaar prepared methyl-*5*-heptatriene-*1,3,5* (octatriene) and methyl-*5*-octatriene-*1,3,5* (nonatriene) for synthetical purposes. In order to obtain methyl-*5*-heptatriene-*1,3,5* he produced tiglinaldehyde according to Grignard's and Abelmann's[2]) method and let the mixture of tiglinaldehyde and allyl bromide react in a solution of ether with zinc. Hereby methyl-*5*-hepatriene-*1,5-ol-4* was obtained, a compound having a refreshing odour, reminding rather of menthol. By heating the former with pulverized potassium bisulphate on a sandbath at a pressure of 70 to 100 mm. in a current of carbon dioxide, the desired octatriene is formed, an unstable substance which very easily resinifies and polymerizes. It turned out to be impossible to hydrogenize octatriene by the aid of sodium and alcohol, as it became resinous immediately. When, however, this hydrocarbon was heated with sodium without a solvent a compound resulted which closely resembled artificial rubber.

In order to produce methyl-*5*-octatriene-*1,3,5*, Enklaar started from *α,β*-methylethyl acrolein, prepared according to Grignard's and Abelman's above-mentioned method, and allowed it to react in the same manner as tiglinaldehyde. The resulting methyl-*5*-octatriene-*1,5-ol-4* was then transformed into nonatriene in the same manner as indicated concerning octatriene; the former polymerizes easily and smells rather of aniseed.

Enklaar supplies the refractometrical figures for *allo*ocimene again because those he mentioned formerly were miscalculated. He also reports those of ocimene and of decane which is made from the former, besides those of a few other saturated hydrocarbons.

Finally, he refers to the difficulties he met in performing the combustion of the above-mentioned compounds, recommending the use of lead chromate in order to obviate them.

On the qualities of pure *d*-camphene, *vide* page 147.

G. Komppa and R. H. Roschier[3]) have confirmed by means of a *complete synthesis of α-fenchene* the formula Wallach had advanced for it[4]). They began with rac. *α*-fenchocamphorone (I) which they treated with methylmagnesiumiodide in dry ether. —

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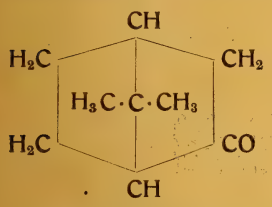
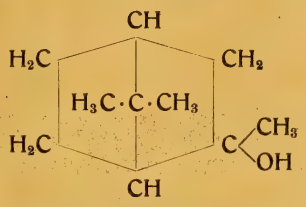
[1]) *Journ. f. prakt. Chem.* II. **84** (1911), 41. — [2]) *Bull. Soc. chim.* IV. **7** (1910), 639. — [3]) *Ann. Acad. Scient. Fennicae*, Series A, vol. X. **3** (1916), 3; *Chem. Zentralbl.* **1917**, I. 751. — [4]) Comp. K. Auwers, *Liebigs Annalen* **387** (1912), 240; *Report* April **1912**, 168.

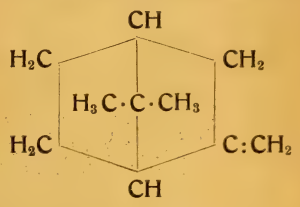
The hydrolysis of the product of the reaction gave an alcohol (II) which, when distilled under atmospheric pressure, split off water, forming thereby rac. α-fenchene, boiling between 154 and 156°; $d_{4^\circ}^{20^\circ}$ 0.8660; n_{D20° 1.47045. They first took it for *iso*-pinene[1]) owing to its identical qualities. However, the action of ozone on α-fenchene gave absolute proof of its constitution, as hereby rac. α-fenchocamphorone semi-carbazone, m. p. 220°), as well as a monobasic acid $C_{10}H_{16}O_2$, melting at 105°, rac. α-fenchenylanic acid, were the resulting products. By way of comparison the authors prepared *iso*pinene from *cyclo*fenchene hydrochloride; its oxidation with potassium permanganate produced rac. hydroxy-α-fenchenic acid (m. p. 139 to 140°) gave no depression of the melting point with the hydroxy-acid prepared from rac. α-fenchene, accordingly no fenchenonic acid had been formed. The action of ozone produced the



(I) α-Fenchocamphorone.



(II) Alcohol, reaction product of fenchocamphorone and methyl-magnesiumiodide.



(III) α-Fenchene.

same compounds. Besides this, the hydrochlorides of *iso*pinene and of the synthetical rac. α-fenchenes are identical. The fenchenonic acid obtained by Aschan[2]) from *iso*-pinene would have been according to Komppa and Roschier rac. *d,l*-hydroxy-α-fenchenic acid polluted with *apo*camphoric acid. That this mixture splits off bromoform under the influence of sodium hypobromite, as Aschan observed, was probably based on an error, because fenchocamphorone, in connection with the smell of the hypobromite, produces a smell resembling bromoform.

Rac. α-fenchene, obtained according to Wallach's method, when treated with ozone, gives rac. α-fenchocamphorone and rac. α-fenchenylanic acid.

The hydrochloride of the rac. α-fenchene can easily be obtained by the action of hydrochloric acid on rac. α-fenchene, prepared according to Wallach's method. M. p. 35 to 37°; b. p. 81° (12 mm.). No depression of the melting point takes place when it is in contact with the hydrochloride of *iso*pinene.

To judge from the fact mentioned above, the term "*iso*pinene" for the hydrocarbon obtained from pinene hydrochloride ought to be abolished. It is not apparent, however, from the abstract if Komppa and Roschier have expressed themselves in this sense. At the time that Zelinsky called the hydrocarbon prepared from pinene by means of reducing substances "*iso*pinene", he had no right to do so, as this term had already been applied to another compound. Accordingly, the strange case would present itself that a term which had been disposed of without any right should justify its existence owing to subsequent investigations.

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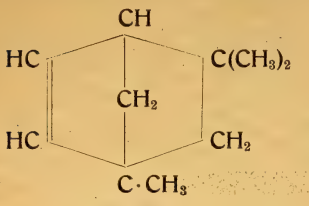
[1]) *Iso*pinene is a hydrocarbon which, according to Aschan, is produced from pinolene hydrochloride by splitting off the acid. Aschan obtained pinolene from the mixture of liquid chlorides resulting from the preparation of pinene hydrochloride by decomposition with alkali (*Berl. Berichte* **40** [1907], 2250; *Report* October **1907**, 137; *vide* also *Report* October **1909**, 169). A quite different substance is the *iso*pinene described later on by N. Zelinsky which is prepared by treating pinene with palladium black and hydrogene (*Berl. Berichte* **44** (1911), 2782; *Report* April **1912**, 172. *Vide* also *Über die Totalsynthese des Isopinens, Finnische Akademie der Wissenschaften, Protokoll der Plenarsitzung vom 4. Oktober 1916*). — [2]) *Öfversigt af Finska Vetenskaps Societetens Förhandling* **51**, I. Afd. A, Nr. 9; *Report* October **1909**. 169.

Fenchylene. — S. S. Nametkin and A. K. Rushenceva[1]) have prepared a new synthetical terpene, fenchylene, by heating methyl*iso*fenchyl xanthate up to 230°. It is a liquid posessing a characteristic odour reminding one of fenchene; b. p. 139 to 140° (760 mm.); $d_{4^\circ}^{20^\circ}$ 0.8381; $[\alpha]_D$ —68,76° (in alcohol); n_{D20° 1.4494. Fenchylene easily adds bromine, forms a crystalline nitrosochloride and produces, when oxidized with permanganate in a solution of alkali, *cis*-fenchocamphenic acid.



Fenchylene.

Iso*fenchylene.* — W. Qvist[2]) obtained from *iso*fenchylalcohol by means of Tschugaeff's xanthogenate method a new hydrocarbon in an almost pure state which he terms *iso*fenchylene. The same process was employed for the preparation of *d,l*-fenchene. No remarks are made on the derivate and qualities of *iso*fenchylene.

Polymerization of pinene. — Some time ago L. G. Gurvitsch[3]) observed that "floridin"[4]) acts in a polymerizing manner on pinene. The addition of 5 per cent. floridin to pinene causes violent ebullition, if more is added, even the whole contents of the flask may be blown out. Pinene which had been carefully purified (b. p. 154.8 to 157.9°; $d_{18^\circ}^{18^\circ}$ 0.8627; $[\alpha]_D$ —39.6°) produced, on being treated with 25 per cent. floridin, a liquid of the specific gravity of 0.8928 (at 18°); $[\alpha]_D$ —5.0°. This gave off 6 to 7 per cent. of camphene on being fractionated; dipentene had not been formed, but 37 per cent. of the resulting liquid seemed to consist of sesqui- and polyterpenes.

Subsequent investigations lead Gurvitsch[5]) to conclude that polyterpenes are the first products of the reaction between floridin and pinene which afterwards are split up into simple terpenes under the influence of the heat of the reaction. The formation of polyterpenes is said to be explained by the absorption of the pinene on the surface of the floridin, whereby the molecules are brought quite close to each other and thus combine producing polyterpenes.

Transformation of pinene into limonene. — H. J. Prins[6]) describes after a detailed introduction, principally of theoretical interest, the transformation of α-pinene into *l*-limonene. For this purpose he mixed 190 g. of *l*-α-pinene from French oil of terpentine (b. p. 156 to 158°; α_D —37°) with 90 g. of glacial acetic acid, added 6 g. of phosphoric acid (d 1.7) to the mixture besides about 5 g. of finely powdered pumice stone and boiled the whole for half an hour. He poured the product of the reaction into water, agitated it with a solution of soda and then distilled it with steam. The substance thus purified yielded, on fractionating it, 107 g. of an oil boiling from 175 to 176° (α_D —34.30°), which the author regards as active limonene. Apart from the very low rotation and from the boiling point he adduces no other constants and has prepared no derivatives.

Although the boiling point corresponds to that of limonene or dipentene, we opine that he has not given sufficient proof of the formation of limonene in reality;

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[1]) *Journ. russ. phys. chem. Ges.* **48** (1916), 450; *Journ. Soc. chem. Industry* **36** (1917), 304. — [2]) *Chem. Ztg.* **41** (1917), 347. — [3]) *Journ. russ. phys. chem. Ges.* **47** (1915), 827; *Journ. Soc. chem. Industry* **34** (1915), 1234. — [4]) In working up the hight-boiling components of petroleum for light lubricating oils one uses decolorants, consisting of aluminium-magnesiumhydrosilicate and termed Fuller's earth or floridin (Muspratt, *Chemische Technologie, Ergänzungsband, 1. Halbband* 1917, 63. — [5]) *Journ. russ. phys. chem. Ges.* **48** (1916), 337; *Journ. Soc. chem. Industry* **36** (1917), 304. — [6]) *Chem. Weekblad* **13** (1916), 1264.

for this purpose the preparation of at least one derivative, for instance of the tetrabromide, would be imperative; to judge from the low rotation it might only be a case of dipentene.

Besides this, the author reports on the polymerization of α-pinene by treating it with aluminium chloride or other metallic chlorides, whereby polyterpenes are formed, and on addition products of pinene. After heating α-pinene for an hour in benzene with aluminium chloride at 30 to 40°, he obtained a mass resembling colophony. When less time is employed, a diterpene is also formed. If pinene is heated with acetic acid and α-naphthalenesulfonic acid for 3 hours about 30 per cent. of diterpene is the result. The use of formic acid improves the yield.

The information on addition products of pinene is purely of a theoretical nature.

Optical rotation of pinene hydrochloride. — According to older data[1]) the so-called pinene monohydrochloride has certain strange characteristics from an optical point of view, since the hydrochloride prepared from dextrorotary oil of turpentine is said to be inactive, whereas that obtained from lævorotatory oil of turpentine is said to be lævorotatory. D. E. Tsakalotos and B. Papaconstantinou[2]) were unable to confirm this unusual phenomenon, on the contrary they obtained from dextrorotatory oil of turpentine a pinene hydrochloride which rotated in the same sense. They worked up the portion of some Greek oil of turpentine boiling from 155 to 156° (760 mm.), which gave the constants $[\alpha]_D$ + 45.1 to + 47.7° and can be regarded as pure α-pinene. By means of passing dry hydrochloric acid gas through dry pinene, cooled by the help of ice, they obtained a pinene hydrochloride which they purified by recrystallizing from alcohol. The rotatory power was determined in acetone and in alcohol: $[\alpha]_D$ + 32.92° at 15° (in acetone) up to + 33.43° at 14.5° (in alcohol), on the average + 33.19°. This *d*-pinene hydrochloride melts at 127° and boils at 207° (750 mm.).

The authors also prepared *d*-pinene hydrochloride in the same fashion: $[\alpha]_D$ + 31.31° at 21° in alcoholic solution. Ilts melting point is at 80° and it boils with partial decomposition.

In order to obtain pure *d*-camphene, they heated *d*-pinene hydrochloride with sodium phenolate. Pure *d*-camphene melted at 42.7° and boiled at 157.6° (745 mm.): $[\alpha]_D$ + 83.5° at 25° (10 per cent. solution in alcohol), + 84.05° at 28° (10 per cent. solution in benzene), + 83.8° at 27° (20 per cent. solution in acetone).

Cymene. — Under the heading "Dyes from waste sulphite liquor" an article was published in the morning edition of the *Frankfurter Zeitung,* dated April 22nd, 1917, according to which on the authority of the *Papier-Zeitung, Berlin* the problem had been solved of producing dyes from sulphite liquor which is a waste product in the manufacture of paper[3]), with the same facility as this is done in Germany from coal tar. The cymene[4]) contained in the sulphite liquor was said to be the raw material for making such dyes. It would further be possible in Scandinavia to obtain about 300 000 kg. of this hydrocarbon from the waste liquor of the paper mills, by means of which, according to a fairly simple process, cotton and woollen goods could by dyed yellow or red, more complicated methods also allowing the manufacture of other coloured dyes.

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[1]) Comp. Wallach, *Terpene und Campher, 2. Aufl.,* Leipzig 1914, p. 238. — [2]) *Journ. de Pharm. et Chim.* VII. **14** (1916), 97. — [3]) Concerning the by-products of the manufacture of cellulose according to the sulphite process comp. page 74 of this *Report.* — [4]) On the formation of cymene in the sulphite cellulose process comp. *Report* April **1901**, 67.

In connection with the above, a paper published by E. Boedtker and O. M. Halse[1]) on the Friedel-Crafts reaction attracts interest. They draw attention to the fact that hitherto no use has been found for the *p*-cymene which is formed as a by-product in the manufacture od cellulose, so that it has been the custom to let it escape into the air. The raw material contains about 90 per cent. of pure cymene. It is quite feasible to convert this in cumene or toluene which are the starting materials for manufacturing dyes.

They boiled 150 g. of crude cymene with 1 kg. of benzene and 30 g. of aluminium chloride for 6 hours on the water-bath and thus obtained 52 g. of toluene, 75 g. of cumene, 6 g. of unchanged cymene, and 20 g. of compounds boiling above 200°. In a second experiment, 100 g. of cymene, dissolved in 1 kg. of benzene, produced after 6 hour's boiling on the wather-bath in the presence of 20 g. of aluminium chloride 41 g. of toluene and 85 g. of cumene, whereas the residue weighed 15 g. In a third experiment, the authors heated 100 g. of cymene, 1 kg. of benzene and 10 g. of aluminium chloride for 8 hours on the water-bath. The results were: — 31 g. of toluene, 67 g. of cumene, whereas 10 g. of cymene remained unchanged. Finally, 90 g. of cymene, 900 g. of benzene and 4.5 g. of aluminium chloride were boiled for 10 hours on the water-bath, whereby 44 g. of toluene and 68 g. of cumene were formed and the residue only consisted of 3 to 4 g.

Alcohols.

Mentholglucosides. — *β*-Glucosides of terpene alcohols have already been described previously. For instance, E. Fischer and K. Raske[2]) have prepared the *β*-glucosides of menthol and borneol; subsequently J. Hämäläinen[3]) described the *β*-glucoside of borneol anew and also obtained a large number of glucosides of other terpene alcohols. Only recently, however, E. Fischer and M. Bergmann[4]) succeeded in the preparation of the *α*-glucoside of a terpene alcohol. They have demonstrated that when acetobromoglucose, phenol, and quinoline are heated together in fairly equal quantities the tetracetyl compound of the already known *β*-phenolglucoside and that of the formerly vainly sought-for *α*-compound are produced. This method can be applied to the hydroaromatic alcohols, for instance to menthol, and by this means the preparation of the hitherto unknown *α*-mentholglucoside has been realized in a very simple manner. A closer investigation of this process has proved that besides the tetracetyl compounds of both the isomeric glucosides also their triacetyl derivatives are produced which can be separated in a pure state by suitable crystallization methods. Apparently, the process of their formation is inseparable from the splitting off of acetyl from the acetobromoglucose, although the work was always undertaken with materials which were as dry as possible; hence the splitting-off of these acetates taking place so readily can be easily explained. This secondary reaction can even proceed still further, in fact to the formation of di- and mono-acetates of the corresponding glucosides. This circumstance may cause considerable loss, but the damage can easily be made good by means of reacetylation with pyridine and acetic anhydride.

α- and *β*-Menthol glucoside behave in the same manner which characterizes this class of compounds towards yeast enzymes and emulsin, or rather as one says nowadays towards *α*- and *β*-glucosidase. There is also a difference between them in the same sense as between *α*- and *β*-methylglucoside, but quantitatively it is much less great.

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[1]) *Bull. Soc. chim.* IV. **19** (1916), 444. — [2]) *Berl. Berichte* **42** (1909), 1465; *Report* October **1909**, 178. — [3]) *Chem. Zentralbl.* **1913**, I. 1604, 1925, 1932; **1913**, II. 869; *Report* October **1913**, 138. — [4]) *Berl. Berichte* **50** (1917), 711. Comp. also E. Fischer u. L. v. Mechel, *ibidem* **49** (1919), 2813.

The authors were also able to elaborate a simple method for preparing α-menthol-glucoside, so that it is by far the easiest to produce of all artifical glucosides of the aromatic and hydroaromatic series.

When acetobromoglucose is allowed to react on *l*-menthol in the presence of quinoline at a temperature of 100 to 105° one obtains a mixture of tetracetyl compounds and of derivatives of α- and β-mentholglucoside which are more deficient in acetyl. In order to reacetylate the latter completely one dissolves the raw material in pyridine, adds the same quantity of acetic anhydride at a low temperature and lets it stand for 24 hours at room temperature. Hereupon one pours the product of reaction into ice water, dissolves the liberated oil in ether, frees the etheral layer from pyridine by agitating it with diluted sulphuric acid, from acetic acid by means of bicarbonate solution, and finally evaporates it after washing it with water. The tetracetyl-β-menthol-glucoside separates itself from the viscous residue in a crystalline state. Tetracetyl-α-mentholglucoside remains in the mother liquor and can be eliminated by means of an inoculation crystal (to be obtained from the triacetyl compound). Melting point 82 to 83°.

As mentioned above, the reaction of acetobromoglucose with menthol in the presence of quinoline also produces, besides the two tetracetyl compounds, substances more deficient in acetyl of which the triacetyl compounds of α- and β-menthol glucoside can be isolated without much trouble. Triacetyl-β-mentholglucoside melts at 143 to 146°, the α-glucoside at 99 to 100°.

α-*l*-menthol-*d*-glucoside is formed from the above-mentioned acetyl derivatives by saponification with alkali in watery-alcoholic solution. Its preparation in a pure state is very simple owing to its slight solubility in water. Its melting point in anhydrous state is between 159 and 160° and it crystalizes out of water in large, thin square leaves.

Owing to its slight solubility in water, the glucoside can also be easily prepared without any intermediate products. Acetobromoglucose is heated with menthol and quinoline for 2 hours at 100°, the mass is then dissolved in water and ether and the etheral layer washed first with diluted sulphuric acid and than with bicarbonate. After the ether has been evaporated one drives off the excess of menthol by the aid of steam. The residual brown-red oil is easily separated from water when it has cooled down. It is dissolved in warm alcohol, mixed at about 60° with seminormal caustic potash solution, and the clear liquid then maintained for about 10 minutes at the same temperature. On adding water the crystallization of the α-mentholglucoside begins immediately, whereupon one cools the liquid down to 0° for about an hour and filters it. Recrystallizing it once from diluted alcohol suffices to produce a quite pure substance.

Owing to the slight solubility of the glucosides in water one can easily hydrolyze their solutions by heating them with a mixture of glacial acetic acid and seminormal hydrochloric acid in a sealed tube.

Sabinol.—Owing to the fact that very differing constants are mentioned in current literature concerning sabinol and that it contains 3 asymmetrical carbon atoms which presupposes the existence of 8 isomerides, V. Paolini and G. Rebora[1]) have attempted to separate them. They transformed sabinol which originated from the fractions of saponified savin oil boiling from 200 to 215°, into its hitherto unknown phthalic acid derivative, a substance crystallizing in white needles and melting at 94 to 95° (from

[1]) *Atti R. Accad. dei Lincei*, Rome (5) **25** (1916), II. 377; *Chem. Zentralbl.* **1917**, I. 752.

benzine); $[\alpha]_D$ —14° 63′ (in methylalcohol; c = 7.382). The acid phthalate produced on saponification pure sabinol, boiling at 208°; $d_{15°}$ 0.9518; $[\alpha]_D$ +7° 56′; $n_{D18°}$ 1.4895. The strychnine salt of acid phthalic sabinol ester forms colourless needles melting at 200 to 201°. This investigations proved that savin oil contains no other isomeric sabinols.

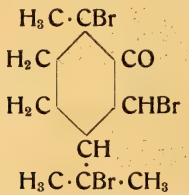
Ketones.

Condensation of methylethylketone with benzaldehyde. — Some times ago, already Harries and Müller[1]) demonstrated that methylethylketone condenses with benzaldehyde in the presence of alkali, forming thereby an α-benzylidene derivative, whereas in the presence of muriatic acid a γ-benzylidene derivative is produced. Accordingly to their opinion the α-compound forms with benzaldehyde a further condensation product, namely a pyrone compound, $C_{18}H_{18}O_2$, melting at 68.5°. H. Ryan and A. Devine,[2]) on repeating these experiments, indeed obtained the same α-benzylidene compound, but instead of the substance $C_{18}H_{18}O_2$, melting at 68.5°, they got an isomeric compound or the same one in a purer state, as it gave a melting point of 81 to 83°. They regard it as an unsaturated ketone and not as a pyrone derivative because it produces an orange colouring with concentrated sulphuric acid.

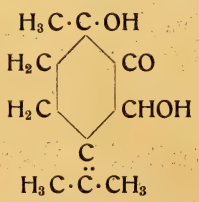
Methylethylketone condenses with piperonal (heliotropine) in a solution of alcoholic hydrochloric acid, producing a blue substance (an oxonium-salt?) which forms with alkali a compound $C_{28}H_{20}O_7$, melting at 212°.

On the use of methylheptenone as an insecticide vide page 138 of this *Report*.

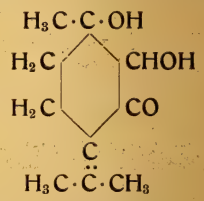
Transformation of tribromotetrahydrocarvone into dihydroxycitronellic acid. — G. Cusmano[3]) described some time ago, in common with P. Poccianti, the transformation of dibromotetrahydrocarvone into buchu camphor, whereby he assumed the formation of two unsaturated intermediate products which he imagined were changed by means of mutual reduction and oxidation between the double bond and the secondary carbinol into a ketone tautomeric to buchu camphor. Cusmano[4]) has now attempted to produce similar



(I) Tribromtetrahydrocarvone.



(II) Hypothetical intermediate product.



(III) α,β-Dihydroxycitronellic acid.

unsaturated carbinols by starting from halogenized terpene ketones whose halogen he removed by means of alkali, for which purpose the tribromotetrahydrocarvone (I), described by Wallach,[5]) seemed suitable to him. Contrary to his expectations, however, alkali did not effect the formation of a double bond in the nucleus, but instead of that the bromine atoms round the nucleus were substituted by hydroxyl. The resulting compound (II) is then split up either directly or after rearrangement by the alkali into a tautomeric substanc, an acid $C_{10}H_{18}O_4$, an α,β-dihydroxycitronellic acid (III). The

[1]) *Berl. Berichte* **35** (1902), 966. — [2]) *Proc. Roy. Irish Acad.* **32** (1916), 208; *Journ. chem. Soc.* **110** (1916), I. 654. — [3]) *Chem. Zentralbl.* **1914**, II. 481; *Report* April **1915**, 104. — [4]) *Atti R. Accad. dei Lincei*, Rome (5) **24** (1916), II. 520; *Chem. Zentralbl.* **1916**, I. 1146. — [5]) *Liebigs Annalen* **286** (1895), 127.

latter forms methylheptenone (semicarbazone, m. p. 135°) when oxidized with lead oxide and acetic acid or when its sodium salt is heated in a dry state. The reduction of α,β-dihydroxycitronellic acid leads to an acid $C_{10}H_{20}O_4$, the α,β-dihydroxyhydrocitronellic acid whose sodium salt produces on heating methylheptanone (*iso*amylacetone).

On the maximum tension of camphor vapour see page 118.

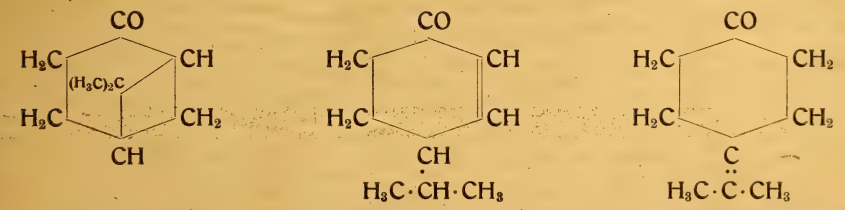
Condensation of p-*tolylidenecamphor with* p-*tolylmagnesiumbromide.* — A. Haller[1]) together with Ramart has studied the effect of *p*-tolylmagnesiumbromide on *p*-tolylidenecamphor, whereby the formation of two isomeric di-*p*-tolylcamphomethanes was to be expected. The viscous condensation product produced, on being distilled *in vacuo* (15 mm.), a fraction boiling between 100 and 240°, from which a crystalline mixture separated itself which by means of repeated crystallization could be resolved into 2 components, namely *p*-tolylidenecamphor (melting point 98°) and *p*-ditolyle (melting point 119 to 120°). Two crystalline compounds were isolated from the portions boiling between 240 and 265° which gave the melting points of 108° and 98 to 99° respectively. The various products of reaction turn, when heated with alcoholic caustic potash, into a compound melting from 113 to 114°.

The substance with the melting point 98 to 99° gave $[\alpha]_D + 75°$, that melting from 113 to 114° $[\alpha]_D$ about $+16°$.

For the time being, the authors regard the compound melting at 108° as a mixture of the two substances melting from 98 to 99° and from 113 to 114° respectively.

Accordingly, the condensation of *p*-tolylidenecamphor with tolylmagnesiumbromide leads to two isomeric di *p*-tolylcamphor methanes, just as that of benzalcamphor with phenylmagnesiumbromide produce two isomeric diphenylcamphormethanes.

Transformation products of nopinone. — In order to explain the relations between polycyclical terpenes to monocyclical ones, their attitude towards sulphuric acid is of paramount importance. In this manner camphor reacts with sulphuric acid producing dihydrocarvone or its product of rearrangement carvenone. According to E. Rimini[2]), nopinone (I), a ketone, which besides by other means can be prepared from nopinic



(I) Nopinone. (II) *Iso*propyl-Δ^2-hexanone-4. (III) *Iso*propylidene*cyclo*hexanone-4.

acid, an oxidation product of β-pinene, by oxidation with lead peroxide or permanganate in a solution of sulphuric acid, produces on being treated with concentrated sulphuric acid at room temperature a mixture of *l-iso*propyl-Δ^2-*cyclo*hexanone-4 (II) and of *l-iso*propylidene*cyclo*hexanone-4 (III). When boiled, however, with diluted sulphuric acid only the former originates. This ketone yields a semicarbazone melting at 201°, whereas Δ^2-*iso*propylhexenone semicarbazone melts at 183°. *Iso*propylidene hexanone-4 produces acetone and tetrahydroquinone on being oxidized with permanganate.

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[1]) *Bull. Soc. chim.* IV. **19** (1916), 219. — [2]) *Gazz. chim. ital.* **46** (1916), II. 119; *Chem. Zentralbl.* **1916**, II. 905. — *Journ. chem. Soc.* **110** (1916), I. 655.

Phenolethers.

Polymerides of anethole and of iso*safrole.* — E. Puxeddu [1]) has now continued an investigation with L. Scaffidi [2]) on the polymerides of anethole and *iso*safrole about which he had already furnished some information at a former period. He describes the polymerization of anethole according to 3 methods. On allowing concentrated sulphuric acid to react with anethole he obtained anisoin, generally in an amorphous, occasionally, however, in a crystalline state, melting from 205 to 210°; at the same time a polymeric anethole $(C_{10}H_{12}O)x$, a yollowish crystalline powder melting at about 250° was formed. When anethole is dissolved in acetone and submitted to the action of iodine, anisoin, in the shape of a white amorphous powder melting at 185°, is the result. The reaction of ferric chloride on an etheral solution of anethole leads to an anisoin $(C_{10}H_{12}O)x$, an amorphous powder melting at 210°, besides to a new polymeric anethole $(C_{10}H_{12}O)x$, which was insoluble in ether and consisted of a white powder melting above 300°. The anisoin produced by means of sulphuric acid gave, when treated with ethereal bromine, apparently a mixture of mono- and dibromine compounds, whereas the anisoin prepared with the aid of ferric chloride absorbs much less bromine. The product of the reaction of ferric chloride on an ethereal solution of *iso*safrole is a white crystalline powder, a dimeric, melting at 92°. Its composition — from cryoscopical data — was found to be $C_{20}H_{24}O_4$.

Years ago, we also obtained a polymeric *iso*safrole, melting at 90 to 91°, on heating it with glacial acetic and sulphuric acid according to Bertram's and Walbaum's method. This product boiled at 220° (3 mm.) [3]).

The reduction of azothymol and of azocarvacrol has been described on page 156.

Acids and Esters.

Campholenic acid. — The products of splitting-up of camphor, belonging to the *cyclo*pentane series, namely campholic acid, camphoric acid, and their further transformation products are generated by the disrupture of the bicyclical system between the carbonyl radicle and the neighbouring methylene group. On treating camphoroxime with dilute acids, a scission takes place between the carbonyl group and the neighbouring quaternary carbon atom whereby the oxime loses water and forms an unsaturated nitrile, which can be saponified to optically active α-campholenic acid. The latter can be transformed into the inactive β-campholenic acid by molecular rearrangement.

Recently, J. R. N. van Kregten [4]) produced α-dihydrocampholenic acid (α-campholanic acid) by hydrogenization from α-campholenic acid. He first produced α-campholenic acid according to Tiemann's [5]) method, namely by transforming camphoroxime into α-campholenic nitrile and by saponification of the latter. Instead of using sulphuric acid for separating the acid from the potassium or ammonium salt he employed acetic acid. He then reduced the campholenic acid according to the process of Sabatier and Senderens by passing its vapour, mixed with pure hydrogen, over nickel heated up to 200°.

In order to purify it the product of the reaction was transformed into its sodium salt by means of soda and the latter then oxidized by means of permanganate in a

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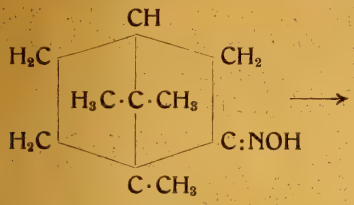
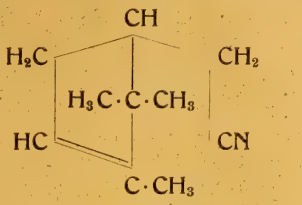
[1]) *Gazz. chim. ital.* **43** (1913), I. 128; *Report* October **1913**, 148. — [2]) *Gazz. chim. ital.* **46** (1916), II. 169; *Chem. Zentralbl.* **1916**, II. 1146. — [3]) *Report* April **1905**, 17. — [4]) *Recueil trav. chim. des P.-B.* **36** (1916), 64; *Chem. Zentralbl.* **1916**, II. 387. — *Journ. chem. Soc.* **110** (1916), I. 480. — [5]) *Berl. Berichte* **29** (1896), 3007.

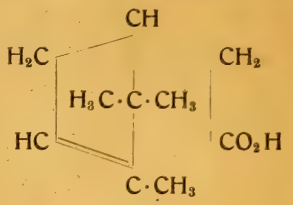
watery solution in order to decompose the remaining campholenic acid; under these circumstances by far the greater part boils from 140 to 142° (12 mm.). However, it was impossible to obtain the acid in quite a pure state according to this method. Accordingly, the author converted the reduced acid by heating its ammonium salt for 5 hours at 220 to 230° into the amide of the α-dihydrocampholenic acid (melting point



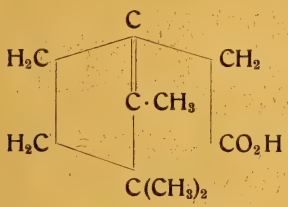
(I) Campheroxime.



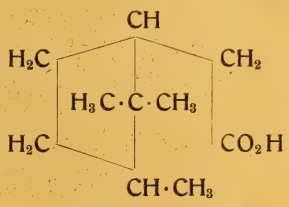
(II) Campholenic nitrile.



(III) Campholenic acid.

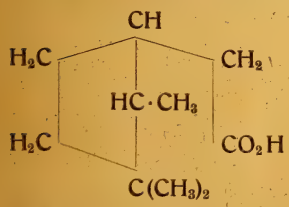


(IV) β-campholenic acid.

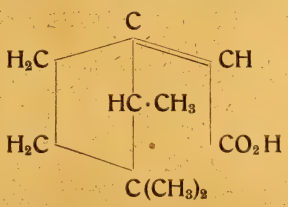


(V) α-dihydrocampholenic acid.

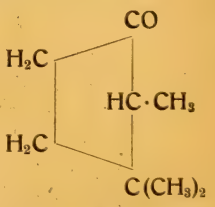
150.5°) which, on being treated with alcoholic caustic potash, turned into the α-dihydrocampholenic acid, boiling from 143 to 144° (12 mm.). Its ethylester forms a pleasant smelling liquid, having the specific gravity 0.9276 $(\frac{14.8°}{4°})$, the anilide melts at 128 to 129°. These data do not tally with those of Mahla and Tiemann[1]) for dihydrocampholenic acid and its derivatives. Van Kregten obtained β-dihydrocampholenic acid (*rac.*-β-campholanic acid) (VI), boiling from 136 to 137° at 10 mm., by reducing β-campholenic acid. It was purified by transforming it into its amide (melting point 157°) and by saponifying the latter in the same manner as described above for α-dihydrocampholenic acid. *Iso*-β-Δ′-campholenic acid (VII) was prepared by treating β-dihydrocampholenic acid with phosphorus pentachloride till the generation of muriatic acid stopped, and



(VI) β-dihydrocampholenic acid (rac. β-campholanic acid).



(VII) *Iso*-β-Δ′-campholenic acid.



(VIII) *1, 1, 2*-Trimethyl-cyclopentanone-3.

hereupon by heating the reaction product for 2 hours on the water bath. Hereupon bromine was added and the mass then poured at a low temperature into alcohol, whereby the ethylester of the α-bromo-β-dihydrocampholenic acid was formed. By heating the latter with quinoline at 200°, the ethylester of the β-Δ′-campholenic acid was formed which on saponification gave β-Δ′-campholenic acid which melts at

[1]) *Berl. Berichte* **33** (1900), 1929.

94.5 to 95.5°. When *β-Δ'*-campholenic acid is oxidized with dilute alcoholic permanganate solution one obtains *1,1,2*-trimethyl*cyclo*pentanone-*3* (VIII), a liquid smelling of camphor and peppermint and boiling at 167 to 169° (760 mm.). The oxime melts at 105 to 105.5° and smells of camphoroxime; the semicarbazone at 221.5 to 222°. Noyes[1]) had already prepared *1,1,2*-trimethyl*cyclo*pentanone from *α*-hydroxydihydrolauronolic acid and from *α,β,β*-trimethyladipic acid.

Iso*teresantalic acid.*—Years ago already, a solid crystalline acid, namely teresantalic acid, was discovered in the saponification liquor of East Indian sandalwood. While its qualities and reactions have been carefully studied by von Soden and Müller, Semmler[2]) and his collaborators devoted themselves to clearing up its constitution. H. Rupe and W. Tomi[3]) were especially interested in this compound because it is so easy to obtain and shows very strong rotation, so they regarded it as a most welcome test material for their investigations on the correlation between constitution and rotatory power. They intended to split up this acid systematically down to simple ring systems, in order to study the alterations of the optical rotation which would ensue from these proceedings.

Hydrochloric acid acts on teresantalic acid in such a manner that hydrochloroteresantalic acid, an addition product, is formed which by means of alkali can be converted into the lactone of the hydroxydihydroteresantalic acid. The authors then intended to split off hydrochloric acid from the above-mentioned teresantalic acid compound, in order to arrive at an unsaturated acid. However, the compound in question proved to be an unsuitable starting material because it was too easily deprived of its carboxyle group. A far more suitable raw material was found in the methylester of the acid which they obtained from the reaction between teresantalic acid ester (produced from the acid by means of dimethylsulphate and alkali) and hydrochloric acid. The next step, splitting off hydrochloric acid, was only possible by heating with aniline; they thus obtained the ester of a perfectly saturated acid, an isomeride of teresantalic acid. A double bond had not been formed, but rather out of the bicyclical system of hydrochloroteresantalic acid, the tricyclical one of teresantalic acid had been rebuilt. The new acid to which the authors apply the term "*iso*teresantalic acid", differs from the old *n*-teresentalic acid by reason of its very much greater optical rotation and besides by its solubility, the crystal water content of its salts, &c., as the following table demonstrates: —

n-teresantalic acid	*iso*-teresantalic acid
melting point 155°	melting point 141.5°
$[\alpha]_{D20°}$ (in benzene) — 76.60°	$[\alpha]_{D20°}$ (in benzene) — 127.58°
calcium-salt contains $1^1/_2$ mol. of water.	Ca-salt contains $^1/_2$ mol. of water.

It is quite impossible that both acids should be structural isomerides; on the contrary, it seems more likely that the isomerism depends on an alteration in the position in space of the carboxyle group.

When teresantalic acid methylester is treated with hydrochloric acid, two isomeric hydrochloric esters are formed, a solid, well-crystallizing *α*-compound (melting point 68°), and a liquid *β*-compound.

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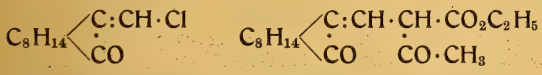
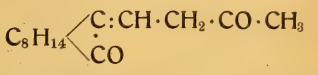
[1]) *Berl. Berichte* **33** (1900), 54. — [2]) Comp. *Report* Oktober **1907**, 87; April **1908**, 93; October **1910**, 129; April **1911**, 105. — [3]) *Berl. Berichte* **49** (1916), 2563.

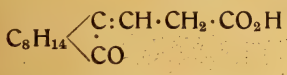
Methylenecamphor acetic acid. — Methylenecamphor offers in the shape of its chloride an easily available and practical starting material for undertaking synthetical work in the camphor group, especially with the object of producing compounds which show a strong rotation. In this manner Rupe and Iselin[1]) prepared a series of derivatives of methylenecamphor from methylenecamphor chloride. Subsequently H. Rupe and E. Burckhardt[2]) attempted to obtain an unsaturated ketone from the same material. They imagined that the reaction of methylenecamphor chloride (I) on sodioacetoacetic acid ester would produce *α*-(camphorylidene-*3*-methyl)-acetoacetic acid ester (methylenecamphor-acetoacetic acid ester) (II) and that one could transform the latter by means of ketone-scission into the ketone (III), namely methyl-(*β*-camphorylidene-*3*-ethyl)-ketone, the one they desired to find. However, the acetoacetic acid ester derivative proved to be very unstable, as when they worked in an alcoholic solution (even to the total exclusion of water), in consequence of an alcoholysis the acid scission took place and the acethyl-radicle was split off as acetic acid ester. When, however, sodioacetoacetic acid ester free from alcohol was employed, the reaction developed in a much more complicated manner.



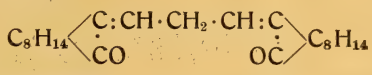
I) methylenecamphorchloride. (II) *α*-(camphorylidene-*3*-methyl)-acetoacetic acid ester (methylenecamphor acetoacetic acid ester).



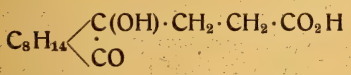
(III) methyl-(*β*-camphorylidene-*3*-ethyl)-ketone.



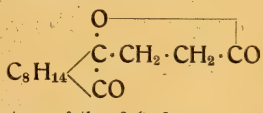
(IV) *β*-(camphorylidene-*3*)-propionic acid (methylenecamphor acetic acid).



(V) *α,γ-di*-(camphorylidene-*3*)-propane (*bis*methylenecamphormethane).



(VI) *β-(hydroxy-3*-camphoryl-*3)* propionic acid.



(VII) lactone of the *β-(hydroxy-3*-camphoryl-*3)* propionic acid.

The easily available main product of the synthesis is the ester of the *β*-(camphorylidene-*3*)-propionic acid or methylene camphor acetic acid (boiling point 171 to 172° at 10 mm.); and from this ester the acid itself (IV) can easily be obtained by saponification with hydrochloric acid. Its melting point is at 106°. Ozone reacts with it producing camphorquinone. A by-product of the reaction of methylene camphor chloride on sodium acetoacetic acid ester is the yellow-coloured *α,γ-bis*-(camphorylidene-*3*)-propane (*bis*methylene camphormethane) (V), melting at 265 to 267° whereby the carboxethyl-group is split off also.

Sodium amalgam does not hydrogenize the double bond of the methylenecamphor acetic acid, on the contrary, the ketone group is attacked whereby a hydroxyacid is formed.

When boiled with caustic alkali solution, methylenecamphor acetic acid forms the salt of the *β-(hydroxy-3*-camphoryl-*3)*-propionic acid (VI), whereas the free acid, on acidulation immediately turns into its lactone (VII), which melts at 207°.

The camphor-radicle most energetically holds fast the double bond of the methylenecamphor acetic acid, in the same manner as in the case of the *β,γ*-unsaturated (*β*-phenylvinyl)-acetic acid, the so-called phenyl*iso*crotonic acid which is the aromatic analogue of methylene camphor acetic acid.

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[1]) *Berl. Berichte* **49** (191), 25; *Report* October **1916**, 123. — [2]) *Berl. Berichte* **49** (1916), 2547.

Methylenecamphor acetic acid reacts in an alkaline solution with diazonium salts, producing strongly-coloured formazyl derivatives; the result of its combination with diazobenzene sulphonic acid is a good dye for wool.

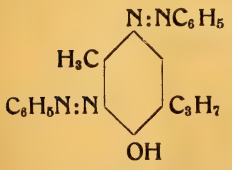
Under the influence of concentrated sulphuric acid, methylenecamphor acetic acid is converted into a new compound, namely a *d*-lactone melting at 66°.

Methylenecamphor acetic acid is easily deprived of its carboxyl group, thereby forming ethylidene-*3*-camphor (methylmethylene camphor), when heated with water under pressure.

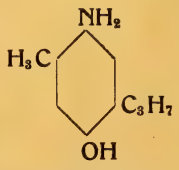
The reaction between methylbenzoate and phenylmagnesiumbromide. — G. Stadnikow[1]), in continuing his investigations on the effects of esters on organic magnesium compounds[2]), has studied the reaction of methylbenzoate on phenylmagnesiumbromide in an ethereal solution free of water, whereby he found that triphenylmethylether and triphenylcarbinol were formed. On allowing benzylbenzoate to act on phenylmagnesiumbromide, dissolved in boiling ether, triphenylmethylbenzylether and triphenylcarbinole resulted. If the process is conducted at a temperature of 100°, one obtains a yield of 15 per cent. triphenylcarbinol and 30 per cent. of triphenylmethylbenzylether. The product of the reaction contains benzylbromide besides. The combination of ethylacetate and of phenylmagnesiumbromide at the boiling point of ether yields α-diphenylethylene. When an ethereal solution of phenylmagnesiumbromide is brought in contact with benzylacetate at a low temperature, the results are methyldiphenylcarbinol and benzylalcohol. The same reaction conducted at the boiling point of ether produces, besides the two above-mentioned compounds, diphenylethylene.

Nitrogenous compounds.

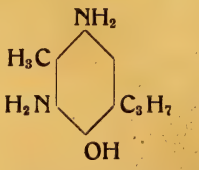
Reduction of azothymol and of azocarvacrol. — While investigating the reducing qualities of phenylhydrazine, E. Puxeddu[3]) also studied the reduction of some diazo compounds. Under these circumstances he found that monoazothymol (benzene-azo-thymol), a substance melting at 112 to 114°, which is the result of the reaction of thymol and of benzenediazoniumchloride, besides the formation of the diazo-compound, produces, when heated with phenylhydrazine, aminothymol (II), melting at 178 to 179°, which latter reacts with ferric chloride, producing thymoquinone. *Dis*azothymol (*dis*benzeneazothymol) (I),



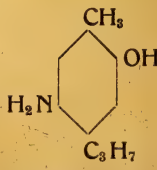
(I) Disazothymol (Disbenzeneazothymol).



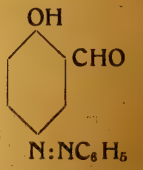
(II) Aminothymol.



(III) Diaminothymol.



(IV) Aminocarvacrol.



(V) Benzeneazosalicylic aldehyde.

a by-product of the preparation of monoazothymol, melts at 180 to 185°, and yields, on being reduced with phenylhydrazine, apparently diaminothymol (III) which, however, is decomposed at 255°, becoming brown at the same time, and is converted, on treatment with ferric chloride, into a hydroxythymoquinone. Monoazaocarvacrol (benzeneazocarvacrol) originates from carvacrol and benzenediazonium chloride at the same time as the diazo compound; it melts at 85° and produces, on being reduced by phenyl-

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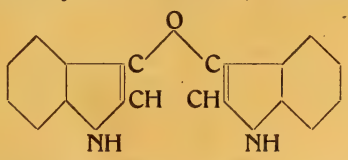
[1]) *Journ. russ. phys. chem. Ges.* **47** (1916), 2037, 2115; *Chem. Zentralbl.* **1916**, II. 388. — [2]) Comp. *Repor* October **1916**, 118. — [3]) *Gazz. chim. ital.* **46** (1916), I. 62, 211; *Chem. Zentralbl.* **1916**, I. 883; II. 471.

hydrazine, aminocarvacrol (melting point 134°) that forms thymoquinone on being oxidized with ferric chloride. *Dis*benzeneazocarvacrol can be reduced to diaminocarvacrol by means of phenylhydrazine, but cannot be isolated; however, oxidation with ferric chloride converts it into β-hydroxythymoquinone melting at 180°. The product of the reaction of salicylic aldehyde and benzenediazoniumchloride is benzeneazosalicylic aldehyde (V) (melting point 132°) which is converted into aminosalicylic aldehyde phenylhydrazone on being reduced with phenylhydrazine.

Autoxidation of α-methylindole under the influence of light.—The important part played by indole in some natural and artificial flower perfumes induces us to discuss a paper by O. Baudisch and A. Hoschek[1]) on the autoxidation of indoles when exposed to full daylight. The experiments were made not only at Bologna, but also at Zürich and Davos, whereby it was most undoubtedly proved that the sun's rays at considerable elevations during winter, as at the Schatzalp Sanatorium (2000 m. above sea level) had a much greater photo-chemical effect than those at Bologna in the height of summer. The authors found that, on exposing a watery suspension of α-methylindole in an atmosphere of oxygen to the rays, apparently in the first instance a red, amorphous substance was formed whose chemical and physical properties indicate that a photo-polymerization of this product had supervened. This red, amorphous compound gradually undergoes a decomposition under the influence of photolysis and oxidation whose primary result is the formation of a yellow substance which proved to be *bis*-[α-methyl-β-indoyl]-ether. Further oxidation converts the latter into *N*-acetylanthranilic acid besides anthranilic acid. Indole itself apparently behaves in the same manner.

Oxidation of indole when exposed to light.—That indole and its derivatives show a tendency towards spontaneous oxidation is a quality of biological importance which has been closely investigated by B. Oddo[2]). He had already had occasion to observe that methylketole (α-methylindole) as well as scatole are superficially transformed under the influence of light into a reddish-yellow substance, insoluble in ether. The compound



which originates from α-methylindole forms yellow crystals, melting at 208 to 209° which evidently contain oxygen. Oddo then exposed indole, scatole (β-methylindole) and methylketole to the light in alcoholic solution with the object of preparing more considerable quantities of these and of analogous compounds. Hereby indole formed a compound $C_{16}H_{12}ON_2$, probably an indoxyl ether of the fellowing constitution. It consists of a yellow powder that melts at 269° with decomp ositionand produces, when boiled with hydrochloric acid, indirubin.

Detection of indole and of scatole.—Some years ago, T. Sasaki[3]) described a sensitive reaction of scatole which is based on the violet colouring that appears when concentrated sulphuric acid is stratified under a methylalcoholic scatole solution. V. E. Nelson[4]) detects

[1]) *Berl. Berichte* **49** (1916), 2579. — [2]) *Gazz. chim. ital.* **46** (1916), I. 323; *Chem. Zentralbl.* **1916**, II. 577. — [3]) *Biochem. Zeitschr.* **23** (1910), 402; **29** (1910), 395; *Report* October **1910**, 203; April **1911**, 192. — [4]) *Journ. of biologic chem.* **24** (1916), 527; *Perfum. Record* **7** (1916), 252.

the presence of indole and of scatole in perfumes in a similar manner. He substratifies an emulsion containing scatole, to which a few drops of dimethylaniline have been added in a test tube with concentrated sulphuric acid; a violet ring is then formed on the surface of contact which is still plainly visible even in dilutions of 1:1000000. This violet compound is soluble in chloroform. If one substitutes citrale for dimethylaniline, scatole produces a red, indole a yellow colouring. The following test is also exceedingly sensitive:— 5 ccm. of a dilute indole or scatole solution are mixed with a few drops of a 5 per cent. solution of vanillin in 95 per cent. alcohol and 3 or 4 drops of hydrochloric acid are then added to the mixture. The presence of indole is indicated by a dark orangered colouring, whereas the presence of scatole shows itself only after heating by a dark violet colouring. This reaction is still visible in dilutions of 1:2000000 to 5000000.

The reduction and hydrolysis of 2, 4, 6-*trinitro*-3-tert.-*butyl*-1-*methylbenzene* and of *trinitro*-tert.-*butylxylene* have been described on page 95 of this *Report*.

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